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RESEARCH DIVISION REPORT 4793-67R

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# **EFFECT OF SULFUR IN JP-5 FUEL ON HOT CORROSION OF COATED SUPERALLOYS IN MARINE ENVIRONMENT**

PROGRESS REPORT NO. 1  
APRIL 1, 1967 TO JUNE 30, 1967

BY  
H. T. QUIGG AND R. M. SCHIRMER

PREPARED UNDER CONTRACT N00019-67-C-027 FOR THE  
NAVAL AIR SYSTEMS COMMAND, DEPARTMENT OF THE NAVY  
BY PHILLIPS PETROLEUM COMPANY  
BARTLESVILLE, OKLAHOMA

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PHILLIPS PETROLEUM COMPANY - RESEARCH DIVISION REPORT 4793-67R

Progress Report No. 1

Naval Air Systems Command Contract N00019-67-C-0275

EFFECT OF SULFUR IN JP-5 FUEL ON HOT CORROSION  
OF COATED SUPERALLOYS IN MARINE ENVIRONMENT

by

H. T. Quigg and R. M. Schirmer

S U M M A R Y

An experimental investigation is in progress to determine whether the 0.4 per cent by weight of sulfur allowed in JP-5 fuel is a safe level for the protection of turbine-blade alloys used in high-performance engines.

Specimens of an aluminum-diffusion coated nickel-base alloy (Misco MDC-1 coated Mar M-200) were exposed to vitiated air from the Phillips 2-inch combustor (60 air-fuel ratio) at high pressure (15 atmospheres), high velocity (745 ft/sec), and high temperature (2000 F) with 5-hour sampling periods and test durations of up to 55-hours. Tests were conducted at all combinations of three levels of sulfur in fuel ( $<0.0040$ , 0.040 and 0.40 weight per cent) with two levels of sea salt in air (zero and 1.0 ppm). The use of specimen weight-loss as a measure of hot-corrosion attack was validated by metallographic examination which showed the absence of deep intercrystalline attack.

Exponential equations of weight-loss with time have been developed and statistically-significant effects have been identified by Covariance Analysis at a 95 per cent confidence level. In all comparisons the removal of sea salt from air significantly decreased the relative rate of corrosion and the level of attack; thus, indicating sea salt to be a primary-causative agent of hot corrosion. The effect of sulfur in fuel varied with the presence or absence of sea salt. In the absence of sea salt in air a reduction of sulfur in fuel from the present limit to either 0.040 or  $<0.0040$  weight per cent had no significant effect on the relative rate of corrosion but decreased the level of attack; however, the level of attack in all cases was only slightly greater than cleaning losses for unexposed specimens and may be of little practical significance. In the presence of 1.0 ppm sea salt in air a reduction in fuel sulfur from the present limit to either 0.040 or  $<0.0040$  weight per cent had no significant effect on the relative rate of corrosion or the level of attack. Variability of weight-loss data with this coating-alloy system was greater than with other systems evaluated and reference specimens indicated that either the coating-alloy system or this sample of the coating-alloy system is unstable.

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PHILLIPS PETROLEUM COMPANY

BARTLESVILLE, OKLAHOMA

Progress Report No. 1  
Naval Air Systems Command Contract N00019-67-C-0275

EFFECT OF SULFUR IN JP-5 FUEL ON HOT CORROSION OF COATED SUPERALLOYS

IN MARINE ENVIRONMENT

1. INTRODUCTION

The corrosion of hot-section parts in modern aircraft-turbine engines is one of the factors that determines the time before overhaul. With operation in a marine environment it becomes a major factor in limiting engine life. Various terms have been used to identify this accelerated attack on the superalloys from which hot-section parts are fabricated. We favor the term "hot corrosion" and will use it in this report to indicate the attack by sea salt on superalloys at high temperature.

Considerable metal loss can be sustained by hot-section parts before failure because hot corrosion advances on a broad front. The attack is led by penetration of randomly dispersed light-grey globules of metallic sulfide. The formation of these sulfides is associated with changes, characterized by chromium depletion, in the surface composition of the alloy. Rapid oxidation of the weakened layer of the alloy follows. Because of the prominent band of precipitated sulfides preceding surface oxidation, hot corrosion is frequently identified as "sulfidation". This has focused attention on the sulfur content of the fuel as being the principal causative agent of hot corrosion. If so, hot corrosion could be controlled by fuel specification. More restrictive limitations on the amount of sulfur allowed in aviation-turbine fuels have been proposed for this purpose, particularly for naval operations.

Most specifications for aviation-turbine fuel allow a sulfur content of 0.40 weight per cent. A significant reduction in the sulfur limit would certainly decrease the amount of available fuel. Also, a more restrictive specification carries with it the potential of higher cost, and a modest increase can amount to a substantial sum because of the large volume involved. Therefore, proposals to lower the sulfur content of aviation-turbine fuel must be approached with caution.

Many investigators feel that deposition of sodium sulfate on the metal surface is a normal precursor to hot corrosion. Some have reasoned that sodium from sea salt and sulfur from fuel combine to form the objectionable sodium sulfate. However, compounds other than sodium chloride are present in sea salt. Sea salt contains 11 per cent by weight of sodium sulfate. Control of hot corrosion by reducing sulfur in fuel to remove one of the critical ingredients from the salt-sulfur combination ignores the sulfur in sea salt.

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For clarification, the Naval Air Systems Command has supported our work to determine whether the maximum sulfur content of 0.40 weight per cent, currently allowed in grade JP-5 aviation-turbine fuel, is a safe level for protection of turbine alloys in high performance engines when operated in a marine environment.

In previous studies, reported in detail by Schirmer and Quigg (1), we attempted to simulate the environment in the turbine section of an aircraft engine with respect to temperature, velocity, pressure, and stoichiometry by use of a high-pressure test facility, originally developed for evaluating the combustion characteristics of aviation-turbine fuels. The test programs included the effect of three levels of sulfur in fuel on hot corrosion of six superalloys, one of which was aluminum coated, at five levels of temperature and three levels of sea salt in air. The five hours of test duration used was sufficient for extensive corrosion of all of the uncoated superalloys under some conditions. It was concluded that an order-of-magnitude reduction (0.40 to 0.040 weight per cent) in sulfur content would not decrease hot corrosion, and no change in fuel specification was indicated. The aluminum-diffusion coating (Misco MDC-1) on Inconel 713C resulted in a material immune to attack under the conditions of exposure used during this investigation; therefore, no measure of the effect of sulfur in fuel on the hot corrosion of a coated superalloy was obtained.

Coating of superalloys is one method which has been demonstrated to reduce attack by sea salt. In previous studies, reported in detail by Quigg and Schirmer (2), we attempted to measure the effect of sulfur in fuel on hot corrosion and the extent of protection afforded by coatings by extending the duration of exposure of specimens to cover a range from 5 up to 55 hours. The programs included one superalloy (Inconel 713C) and two different coating-alloy systems (Misco MDC-1 and Misco MDC-9 on Inconel 713C). In these programs three levels of sulfur in fuel ( $< 0.0040$ , 0.040, and 0.40 weight per cent) and two levels of sea salt in air (zero and 1.0 ppm) were used with exposure at the 2000 F conditions. In addition, tests were conducted with 10.0 ppm sea salt in air at three levels of sulfur in fuel ( $< 0.0040$ , 0.40 and 4.0 weight per cent), with one of the coated superalloys. Exponential equations of weight loss with time were developed which permitted evaluating the effects of sulfur in fuel and sea salt in air. These studies showed one case where a reduction of sulfur in fuel from the present limit to 0.040 weight per cent significantly decreased hot corrosion of a coated superalloy, and another case where attack was significantly increased. This indicated that our previous recommendation based upon a study of bare superalloys, could be extended to coated superalloys; however, additional data are required with other coating-superalloy systems and other exposure temperatures before making a final recommendation as to the level of fuel sulfur for protection of superalloys in a marine environment.

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The purpose of our current investigation is to examine an MDC-1 coating-superalloy system at the 2000 F conditions as to the effect of sulfur in fuel on hot corrosion in a marine environment. In previous studies with bare alloys (1) it was found that while a decrease in sulfur content in fuel from 0.40 to 0.040 weight per cent did not decrease metal loss, in the presence of 10.0 ppm sea salt in air, metal loss actually increased with a decrease in fuel sulfur with some superalloys. Mar M-200 was one of the superalloys that showed an inverse effect of decreasing fuel sulfur content on metal loss and it has been chosen as the base alloy for the current investigation. MDC-1 coating, one of the coatings used in previous studies (2), was selected as the coating for the current investigation. Three levels of sulfur in fuel ( $\leq 0.0040$ , 0.04, and 0.40 weight per cent) and two levels of sea salt in air (zero and 1.0 ppm) are included in the program, with exposure at the 2000 F conditions used in previous studies. Test specimens were removed at five hour intervals during 55-hour tests to provide a series of specimens for each combination of sulfur in fuel and sea salt in air. In addition tests have been completed at two levels of sulfur in fuel with 1.0 ppm sea salt in air with three specimens exposed at each of three periods of exposure (15, 30, and 45 hours) to provide a measure of experimental error. An analysis of these data is presented in this report.

## 2. CONCLUSIONS

Experiments have been conducted, using a high-pressure burner rig, to evaluate the effect of sulfur in JP fuel on the hot corrosion of coated turbine-blade materials. The durability of a coating-alloy system had been investigated at a 2000 F test condition, which simulates exposure in a high-performance engine when operated in a marine environment. Also, for reference purposes, data have been obtained in the absence of sea salt.

The use of weight loss by test specimens as a measure of hot-corrosion attack was validated by metallographic examination, which showed corrosion advancing on a broad front without deep intercrystalline penetration. Electro-cleaning in a hot-caustic bath was used to remove accumulated metal-oxidation products and sea-salt residues from the specimens.

Exponential equations and Covariance Analysis were used to permit evaluation of the effects of three levels of sulfur in fuel ( $<0.0040$ ,  $0.040$ , and  $0.40$  weight per cent) and two levels of sea salt in air (zero and  $1.0$  ppm). The following conclusions can be drawn at a 95 per cent confidence level.

- A. In all comparisons, the removal of sea salt from air significantly decreased the relative rate of corrosion and the level of attack; thus indicating sea salt in air to be a primary-causative agent in the hot corrosion of coated superalloys in a marine environment.
- B. In the absence of sea salt in air, a reduction of sulfur in fuel from  $0.40$  to either  $0.040$  or  $<0.0040$  weight per cent:
  - (a) had no significant effect on the relative rate of corrosion
  - (b) significantly reduced the level of hot corrosion attack.
- C. In the presence of  $1.0$  ppm sea salt in air, a reduction of sulfur in fuel from  $0.40$  to  $0.040$  or  $<0.0040$  weight per cent had no significant effect on the relative rate of corrosion or the level of attack.

While statistically significant differences were found in the level of hot corrosion on the Misco MDC-1 coated Mar M-200 specimens the level of attack was very low with all three concentrations of sulfur in fuel, being only slightly higher than cleaning loss for new unexposed specimens and may not represent a practical loss.

Variability of weight-loss for Misco MDC-1 coated Mar M-200 test specimens in this program was greater than in programs with other coating-alloy systems which indicates that either the coating-alloy system or this sample from the system is less stable than other coating-alloy systems evaluated.



### 3. RECOMMENDATIONS

The primary objective of this investigation is to determine whether the 0.4 weight per cent sulfur currently allowed in grade JP-5 fuel is a safe level for the protection of turbine blades in aircraft engines of advanced design.

Programs have been completed which allowed an evaluation of the effect of sulfur concentration in fuel on the durability of bare superalloys (1). It was concluded that a reduction in sulfur content by an order of magnitude, to 0.04 weight per cent, would not reduce hot corrosion significantly. Therefore, it was recommended that no change in the sulfur limit for JP-5 be made to alleviate hot-corrosion attack on turbine blades.

The recommendation was tempered by another to extend this investigation to evaluate the effect of fuel sulfur on the protection afforded by coatings. Those data were sought because superalloys having the most desirable physical properties are more susceptible to hot corrosion and require coatings for satisfactory durability.

Programs have been completed which allowed an evaluation of the effect of sulfur concentrations in fuel on the durability of two coating-alloy systems (2). These two systems were composed of two different coatings on the same base superalloy. Our analysis of the data showed one case where a reduction of sulfur in fuel from the present limit to 0.04 weight per cent significantly decreased hot corrosion and another case where attack was significantly increased. In the current program, the investigation was extended to include another coating-alloy system composed of one of the coatings from the previous investigation and another base superalloy. It was found that a reduction in fuel sulfur from the current specification maximum to 0.04 weight per cent significantly reduced the level of corrosion in one case and had no effect in another. This indicated that our previous recommendation based upon a study of bare superalloys, can be extended to coated superalloys.

It is emphasized that additional data are required with other coating-superalloy systems and other exposure temperatures before making a final recommendation.

#### 4. RESULTS AND DISCUSSION

This experimental investigation was designed to permit comparisons showing the effect of sulfur concentration in JP-5 fuel on the hot corrosion of a coated superalloy in a marine environment. To characterize the principal levels of interest, data were obtained with three concentrations of sulfur in the fuel (0.40, 0.040, and <0.0040 weight per cent), representing specification maximum, production median, and essentially sulfur-free fuels. A marine environment was simulated by the addition of sea salt to the air (1.0 ppm). Test specimens used were Misco MDC-1 coated Mar M-200. Misco MDC-1 coating is rich in aluminum and is applied by a pack-diffusion process.

In previous studies (1) aluminum coated specimens (Misco MDC-1 on Inconel 713C) were immune to attack under all combinations of temperature, sulfur in fuel, and sea salt in air with the standard 5-hour test duration. In further studies (2) experiments were designed to stress the coating to failure by extending test duration and, thereby, permit evaluating the effect of sulfur in fuel on the durability of coated superalloys. Using a 1-hour cyclic procedure, test specimens were removed, with replacement, at 5-hour intervals to obtain a series of specimens with exposures of up to 55 hours duration.

To simulate the environment in the turbine section of an aircraft engine, a cascade holder supporting six test specimens was mounted in the exhaust section of Phillips 2-inch combustor. The facility was operated to obtain exposure of the test specimens at high pressure (15 atmospheres), high temperature (2000 F), high velocity (745 feet per second), and realistic stoichiometry (60 air-fuel ratio).

Further details concerning the test equipment, test materials, and test program are presented in Appendices 1, 2, and 3, respectively, which are Sections 8, 9, and 10 of this report.

There is no generally-accepted criterion at the present time for determining the effective life of a coating on a superalloy. Evaluations have been based on weight loss by specimens, visual appearance of specimens, and combinations of both.

Whitfield and Parzuchowski (3) established a weight loss of 0.100 grams as the criteria for failure of coated specimens. Using dimensions of the corrosion bars given by Danek (4), the surface area of their specimens is calculated to be 20.00 cm<sup>2</sup>. From these figures we calculated the criteria for failure of the coating to be 5.0 mg/cm<sup>2</sup>.

In Reference 2 a visual rating system was used for rating the appearance of coated test specimens. It was found that coatings were penetrated on specimens showing "Light Surface Attack" or "Light Edge Attack". A weight loss of 5 mg/cm<sup>2</sup> was in the range of weight losses obtained for

visual ratings of "Light" and it was concluded that  $5 \text{ mg/cm}^2$  represents a reasonable criteria for coating failure.

In this report weight loss, visual ratings and metallographic examination of test specimens will be used to evaluate the effect of sulfur in fuel and sea salt in air on hot corrosion.

#### 4.1. Visual Ratings

Visual ratings of each Misco MDC-1 coated Mar M-200 test specimen are shown in Tables 1 and 2. Using the arbitrary merit values assigned to the visual ratings, described in Appendix 1 (Table 7), a plot of logarithms of visual ratings vs logarithms of the weight losses per unit area was made for the Misco MDC-1 coated Mar M-200 specimens, and is shown in Figure 1. As with the data for the two coating-alloy systems shown in Reference 2, the visual ratings decreased as weight loss increased and  $5 \text{ mg/cm}^2$  is in the range of weight losses for a visual rating of "Light".

To further illustrate the effect on appearance of the exposure variables in this investigation, photomicrographs (2X magnification) of a representative set of Misco MDC-1 coated Mar M-200 specimens are presented in Figure 2. It will be noted that following exposure for 45 hours at the 2000 F test condition the specimens show little or no visible evidence of attack in the absence of sea salt, regardless of sulfur concentration. With the addition of sea salt to the environment, hot corrosion is evident; but, as before, sulfur concentration had no visible effect on the intensity of attack. Also, it should be noted that hot corrosion is not uniform across the face of the specimens, indicating that the attack by corrosive agents more easily penetrated the coating in some areas on the specimens. This forecasts a large experimental error with this coating-alloy system and suggests a lack of uniformity in the coating; both of which were found as discussed in subsequent sections of this report concerning the weight-loss data and metallography, respectively.

The visual rating is a subjective value, while weight loss is a physical measurement that should be associated with a smaller error. Visual ratings group the data at only five levels while the use of weight loss does not require the grouping of data. For these reasons, and in the light of metallographic examination which established the mode of attack and will be discussed later, it was decided that weight loss measurements would be used in evaluating the effects of sulfur in fuel and sea salt in air on hot corrosion.

#### 4.2. Weight-Loss Data

In previous studies (1) of weight loss from hot corrosion, using a 5-hour test, an estimate of the variance was found to be a percentage of the value, and logarithms of weight loss per unit area were used to provide a basis of uniform variance. The standard deviation of the weight-loss data in terms of logarithms (1) was 0.288, which is equivalent to a percentage standard deviation of 94 per cent.

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TABLE 1WEIGHT-LOSS DATA AND VISUAL RATINGS FOR MISCO MDC-1 COATED MAR M-200  
(Zero Sea Salt in Air)

Test Number	Sulfur in Fuel, wt %	Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss		Visual Rating (c)
				mg	mg/cm <sup>2</sup> (b)	
5	<0.0040	2	5	42.4	2.09	VLS
	<0.0040	3	10	31.6	1.56	N
	<0.0040	6	15	44.3	2.18	VLS
	<0.0040	1	20	47.5	2.34	N
	<0.0040	4	25	48.1	2.37	N
	<0.0040	4A	30	38.8	1.91	N
	<0.0040	1A	35	42.0	2.07	N
	<0.0040	6A	40	50.5	2.49	N
	<0.0040	3A	45	89.9	4.44	VLS
	<0.0040	2A	50	50.7	2.50	N
	<0.0040	5	55	43.8	2.16	N
4	0.040	2	5	43.1	2.13	N
	0.040	3	10	44.3	2.18	N
	0.040	6	15	45.7	2.25	N
	0.040	1	20	97.5	4.81	VLS
	0.040	4	25	37.5	1.85	N
	0.040	4A	30	73.5	3.63	N
	0.040	1A	35	42.1	2.08	N
	0.040	6A	40	80.9	3.99	LS
	0.040	3A	45	57.0	2.81	N
	0.040	2A	50	62.3	3.09	VLS
	0.040	5	55	51.3	2.53	N
3	0.40	2	5	44.1	2.18	N
	0.40	3	10	49.5	2.44	N
	0.40	6	15	44.0	2.17	VLS
	0.40	1	20	134.2	6.62	VLS
	0.40	4	25	88.1	4.35	VLS
	0.40	4A	30	78.2	3.86	VLS
	0.40	1A	35	82.5	4.07	VLS
	0.40	6A	40	116.2	5.73	VLS
	0.40	3A	45	83.7	4.13	N
	0.40	2A	50	210.7	10.39	MS
	0.40	5	55	59.7	2.94	VLS

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) Mg times 0.049333 = mg/cm<sup>2</sup>

(c) See Table 3 for rating system.

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TABLE 2

WEIGHT-LOSS DATA AND VISUAL RATINGS FOR MISCO MDC-1 COATED MAR M-200  
(1.0 ppm Sea Salt in Air)

Test Number	Sulfur in Fuel, wt %	Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss		Visual Rating (c)
				mg	mg/cm <sup>2</sup> (b)	
2	<0.0040	2	5	74.9	3.70	VLS
	<0.0040	3	10	98.7	4.87	VLS
	<0.0040	6	15	153.0	7.55	LS
	<0.0040	1	20	114.2	5.63	VLS
	<0.0040	4	25	1246.5	61.49	HS
	<0.0040	4A	30	98.4	4.85	LE
	<0.0040	1A	35	985.0	48.59	HS
	<0.0040	6A	40	4725.1	233.10	HS
	<0.0040	3A	45	305.6	15.08	MS
	<0.0040	2A	50	1342.8	66.24	HS
	<0.0040	5	55	4972.7	245.32	HS
2A	<0.0040	2	15	578.9	28.56	LS
	<0.0040	6	15	84.1	4.15	VLS
	<0.0040	3A	15	78.4	3.87	N
	<0.0040	3	30	69.9	3.45	VLS
	<0.0040	2A	30	61.3	3.02	VLS
	<0.0040	6A	30	265.1	13.08	VLS & LE
	<0.0040	1	45	3155.4	155.66	HS
	<0.0040	4	45	1771.5	87.39	HS
	<0.0040	5	45	3479.7	171.66	HS
6	0.040	2	5	43.7	2.16	N
	0.040	3	10	95.9	4.73	VLS
	0.040	6	15	68.7	3.39	VLS
	0.040	1	20	67.0	3.30	VLS
	0.040	4	25	234.7	11.58	MS
	0.040	4A	30	118.6	5.85	LE
	0.040	1A	35	4043.8	199.49	HS
	0.040	6A	40	72.9	3.60	VLS
	0.040	3A	45	7536.1	371.78	HS
	0.040	2A	50	2259.8	111.48	HS
	0.040	5	55	4047.2	199.66	HS

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) Mg times 0.049333 = mg/cm<sup>2</sup>.

(c) See Table 3 for rating system.

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TABLE 2 (Continued)

Test Number	Sulfur in Fuel, wt %	Test (a) Specimen Number	Total Exposure Time, hours	Test Specimen Weight Loss		Visual Rating (c)
				mg	mg/cm <sup>2</sup> (b)	
1	0.40	2	5	44.9	2.22	N
	0.40	3	10	56.5	2.79	N
	0.40	6	15	65.4	3.23	N
	0.40	1	20	106.6	5.26	LS
	0.40	4	25	110.1	5.43	LS
	0.40	2A	25	1972.5	97.31	HS
	0.40	2B	25	148.7	7.34	LE
	0.40	4A	30	317.1	15.64	MS
	0.40	1A	35	80.7	3.98	VLS
	0.40	6A	40	118.9	5.87	LE
	0.40	3A	45	87.8	4.33	VLS
1A	0.40	5	55	98.7	4.87	N
	0.40	2	15	90.8	4.48	VLS
	0.40	6	15	69.9	3.45	N
	0.40	3A	15	278.2	13.72	LS
	0.40	3	30	313.7	15.48	MS
	0.40	2A	30	2916.7	143.89	HS
	0.40	6A	30	1572.4	77.57	HS
	0.40	1	45	5322.1	262.56	HS
	0.40	4	45	6351.7	313.35	HS
	0.40	5	45	3188.4	157.29	HS
1B	0.40	2	15	275.2	13.58	LS
	0.40	6	15	95.8	4.73	VLS
	0.40	3A	15	107.5	5.30	VLS
	0.40	3	30	1786.2	88.12	MS
	0.40	2A	30	220.3	10.87	LE
	0.40	6A	30	3712.2	183.13	HS
	0.40	1	45	3268.9	161.26	HS
	0.40	4	45	4878.4	240.67	HS
	0.40	5	45	2739.0	135.12	HS

(a) Test specimen number indicates position in cascade holder. A letter following the position number indicates a replacement test specimen, following removal of the initial test specimen.

(b) Mg times 0.049333 = mg/cm<sup>2</sup>.

(c) See Table 3 for rating system.

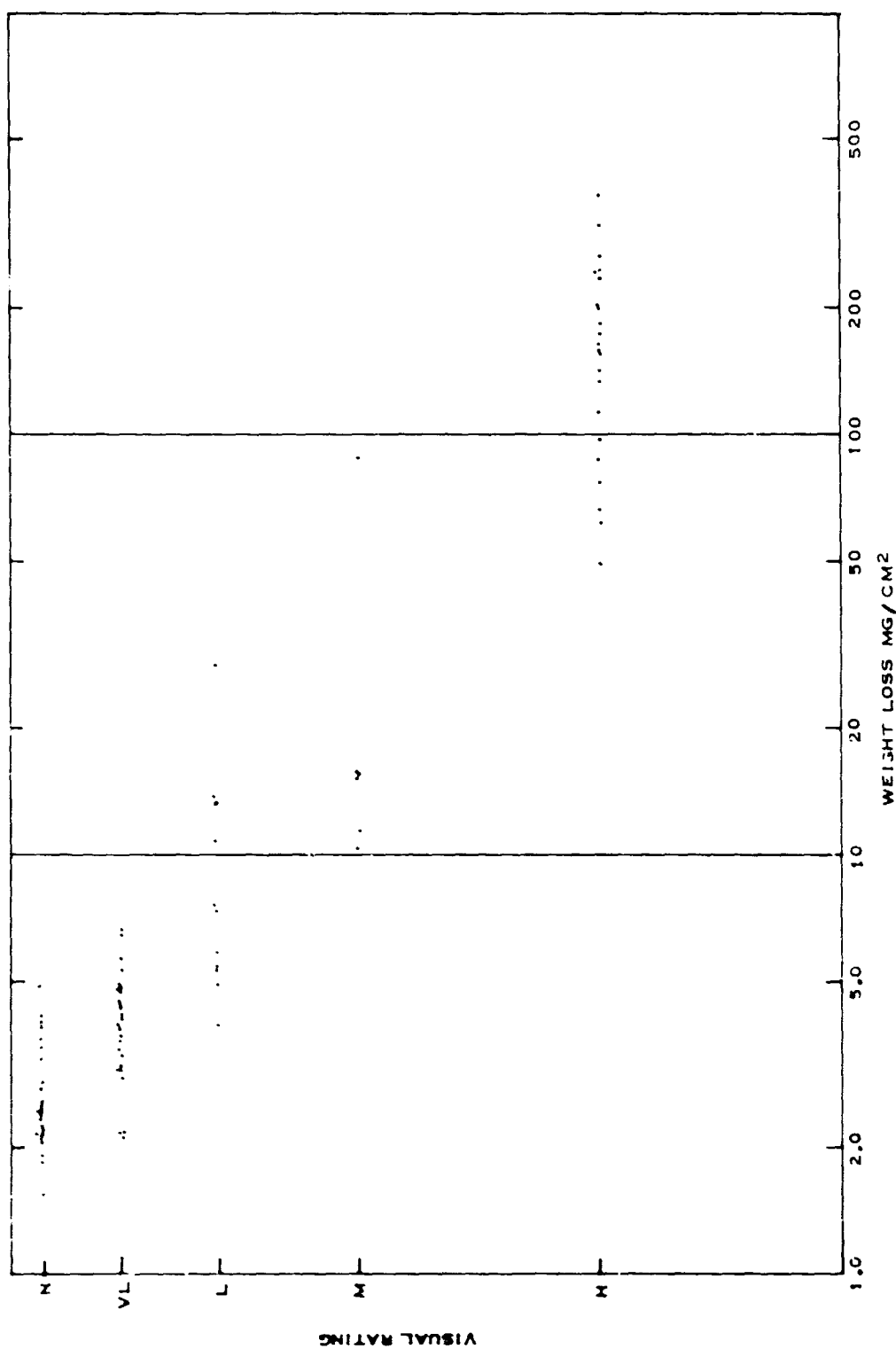
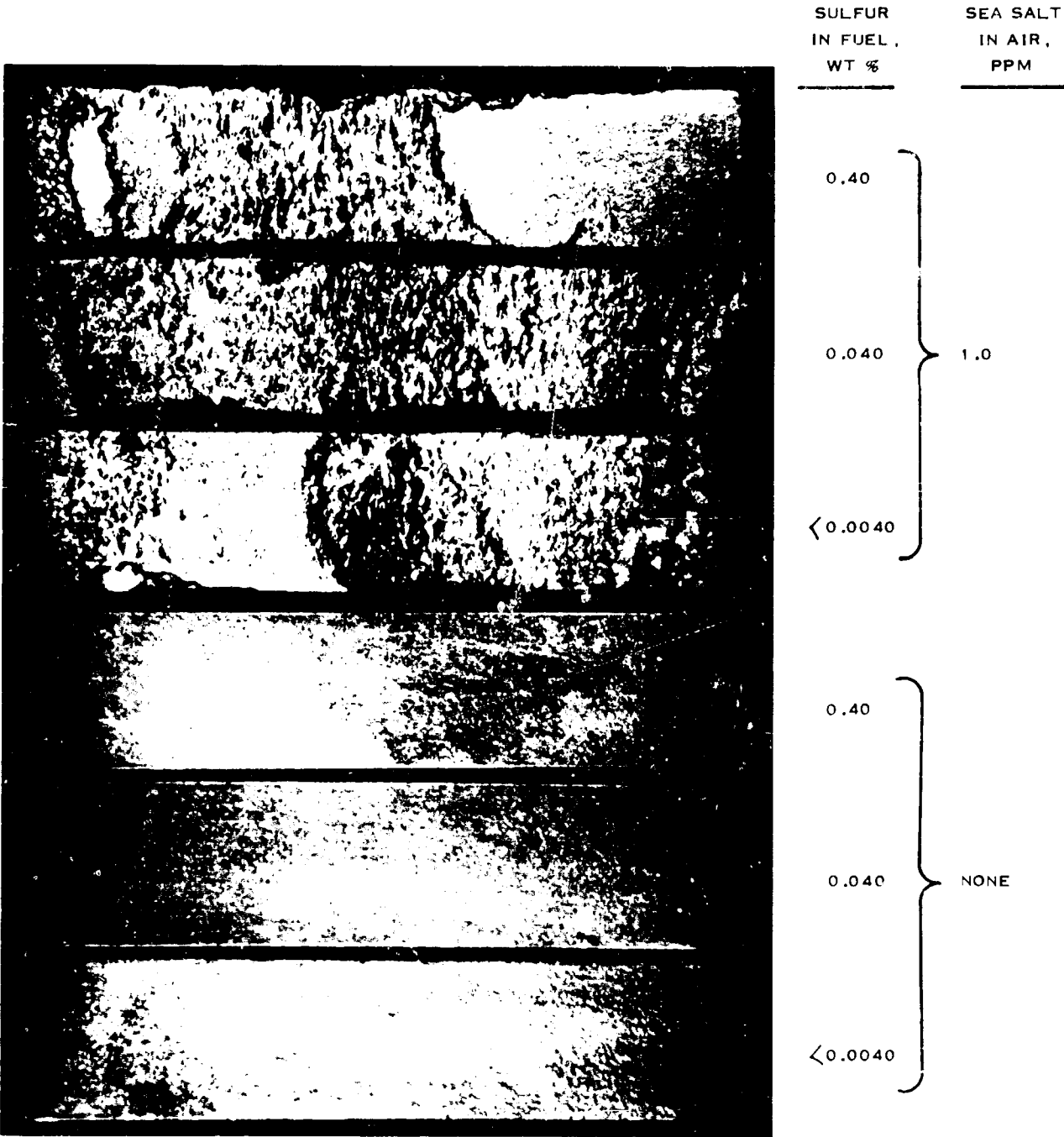


FIGURE 1  
RELATIONSHIP BETWEEN VISUAL RATING AND WEIGHT LOSS  
FOR MISCO MDC-1 COATED MAR M-200





2X MAGNIFICATION AFTER ELECTRO-CLEANING

FIGURE 2  
MISCO MDC-1 COATED MAR M-200 SPECIMENS  
AFTER 45 HOURS AT 2000 F TEST CONDITION

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In the evaluation of the hot-corrosion data for Misco MDC-1 coated Inconel 713C (2) the Standard Error of Estimate (S.E.E.) for the regression equations varied with the level of hot-corrosion attack. These data have subsequently been examined (logarithms of weight-loss) using Bartlett's test for homogeneity of variance (5). A total of 84 specimens representing 30 different combinations of time, sulfur in fuel and sea salt in air were available for comparisons. For all of the data the mean standard deviation was 0.316 in terms of logarithms or 107 per cent; however, the test showed the variance to be non-homogeneous. To further examine the data, an arbitrary separation was made into two groups on the basis of failure or non-failure of the coating as indicated by a weight loss of 5.0 mg/cm<sup>2</sup>. Data for a condition where one or more of the specimens had a weight loss of more than 5.0 mg/cm<sup>2</sup> were placed in the high-weight-loss group (coating failure) and the remaining data were placed in the low-weight-loss group (no coating failure). In the high-weight-loss group there were 64 specimens available from 21 combinations of conditions and in the low-weight-loss group there were 20 specimens available from 9 combinations of conditions. Tests of each group of data indicated homogeneous variance. The mean standard deviation of the low-weight-loss group of data was 0.068 in terms of logarithms (17 per cent) and the mean standard deviation for the high-weight-loss group of data was 0.352 in terms of logarithms (125 per cent). These data will aid in the evaluation of hot-corrosion data from the current program.

In evaluating oxidation with time, many forms of equations have been proposed and used in the literature to express the relationship. These forms include linear, cubic, parabolic, and exponential equations depending upon the system being investigated. In Reference 2 it was shown that Misco MDC-1 and MDC-9 coated Inconel 713C hot-corrosion data were fit by the use of the exponential growth curve which is commonly used in the form:

$$W = (A)(B)^X, \quad (1)$$

where W is weight loss per unit area, X is exposure time, and A and B are constants to be evaluated. Applying logarithms to the equation,

$$\log W + \log A + (\log B) X \quad (2)$$

$$\text{or } Y = a + bX, \quad (3)$$

where  $Y = \log W$ ,  $a = \log A$ , and  $b = \log B$ . Equation 3 is the equation for a straight line. The use of this form of equation permits the use of logarithms of weight loss, which was shown previously to give uniform variance. Data that can be fit by Equation 3 shows that the logarithm of weight-loss increases at a uniform rate over the time-period studied. The slope, b, of Equation 3 is measured in terms of  $\Delta$  logarithms per  $\Delta$  hours, or the ratio of weight losses per hour  $\left[ \frac{(\text{mg/cm}^2)/(\text{mg/cm}^2)/\text{hour}}{(\text{mg/cm}^2)/\text{hour}/(\text{mg/cm}^2)} \right]$ . Thus, b, is the relative rate of corrosion with units of reciprocal hours.

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The weight-loss data for Misco MDC-1 coated Mar M-200 specimens exposed to the three concentrations of sulfur in fuel in the absence of sea salt in air are shown in Table 1 and are presented graphically in Figure 3. The data for each concentration of sulfur in fuel were fit to equations of the form of Equation 3. To evaluate the effect of sulfur in fuel on hot corrosion the data for the three equations were examined using an Analysis of Covariance and the analysis is shown in Table 3. From this analysis it was concluded that there was no significant difference, at the 95 per cent confidence level, among the relative rates of corrosion for the three levels of sulfur in fuel; however, there is a significant difference among the adjusted mean weight losses. To determine which differences were significant 95 per cent confidence limits for differences were calculated and are shown in Table 3. If the confidence limits on a difference do not include zero the difference is statistically significant.

The adjusted means for the three levels of sulfur in fuel are:

Sulfur in Fuel, wt %	Mean, $\log \text{ mg/cm}^2$	Geometric Mean, $\text{mg/cm}^2$
< 0.0040	0.360787	2.29
0.040	0.435113	2.72
0.40	0.597900	3.96

The differences in log weight loss per unit area for reductions in fuel sulfur are:

$$0.40 \text{ minus } 0.040 \text{ wt \%} = 0.597900 - 0.435113 = 0.162787 *$$

$$0.40 \text{ minus } < 0.0040 \text{ wt \%} = 0.597900 - 0.360787 = 0.237113 *$$

These data indicate a statistically significant reduction in weight-loss for a reduction in sulfur in fuel from the specification maximum of 0.40 to either 0.040 or < 0.0040 weight per cent sulfur. This reduction may be of little practical significance because of the very low weight losses in all cases which are only slightly greater than the cleaning losses for new, unexposed specimens of 1.61  $\text{mg/cm}^2$ .

The variability of the data is greater than with previous coating-alloy systems. The standard deviation of the data in terms of logarithms was 0.145 or a percentage standard deviation of 40 per cent. The standard deviation for the Misco MDC-1 coated Inconel 713C low-weight-loss group of data by comparison was only 0.068 in terms of logarithms or a percentage standard deviation of 17 per cent of the value.

The weight-loss data for Misco MDC-1 coated Mar M-200 specimens exposed to < 0.0040, 0.040, and 0.40 weight per cent sulfur in fuel and 1.0 ppm sea salt in air are shown in Table 2 and are presented graphically in Figures 4 to 6. The first test at each of the three concentrations of sulfur

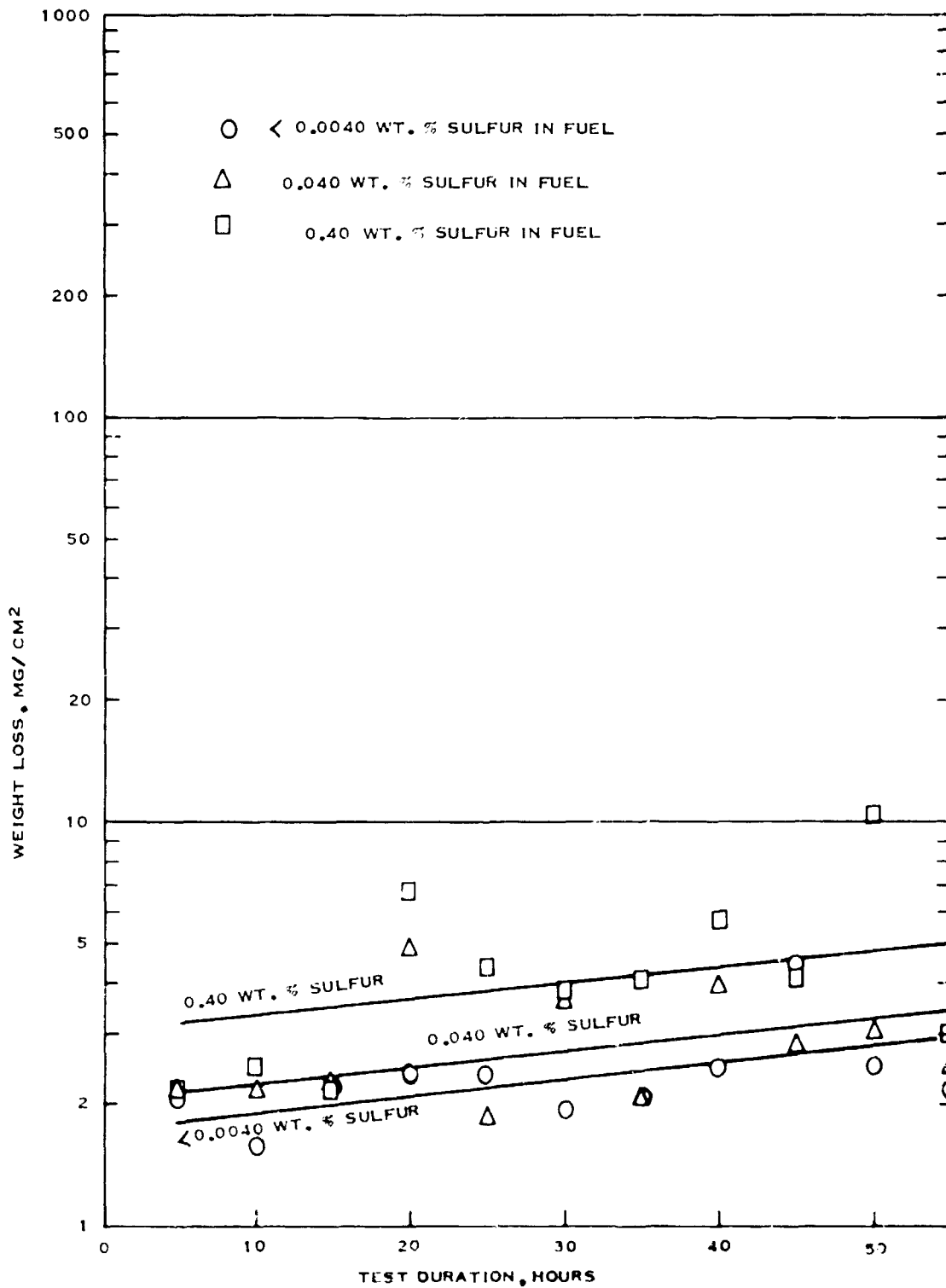


FIGURE 3  
HOT CORROSION OF MISCO MDC-1 COATED MAR M-200 WITH  
ZERO SEA SALT IN AIR

TABLE 3

ANALYSIS OF COVARIANCE OF MISCO MDC-1 COATED MAR M-200 DATA  
(Zero Sea Salt in Air)

Line	df	$\sum x^2$	$\sum xy$	$\sum y^2$	b	Deviations From Regression		
						df	$\sum d_{y..}^2$	Mean Square
1 $\bar{C} = 0.0040$	10	2750.0	9.064115	0.124361	0.003296	9	0.094486	
2 $\bar{C} = 0.040$	10	2750.0	5.732745	0.177540	0.002084	9	0.175675	
3 $\bar{C} = 0.40$	10	2750.0	19.252640	0.449714	0.007000	9	0.314928	
4 Within						27	0.585089	0.021669
5 Reg. Coef.						2	0.025997	0.012998 (-)
6 Common	30	8250.0	34.049500	0.751615	0.004127	29	0.611086	0.021071
7 Adj. Means						2	0.323569	0.161784 (*)
8 Total	32	8250.0	34.049500	1.075184		31	0.934655	

(-) No significant difference among regression coefficients at 95 per cent confidence level.

(\*) Significant difference in adjusted means at 95 per cent confidence level.

$D \pm 0.126575 = 95$  per cent confidence limits on difference between two adjusted means.

$\sqrt{0.021071} = 0.145160$  (40 per cent) = Standard Deviation

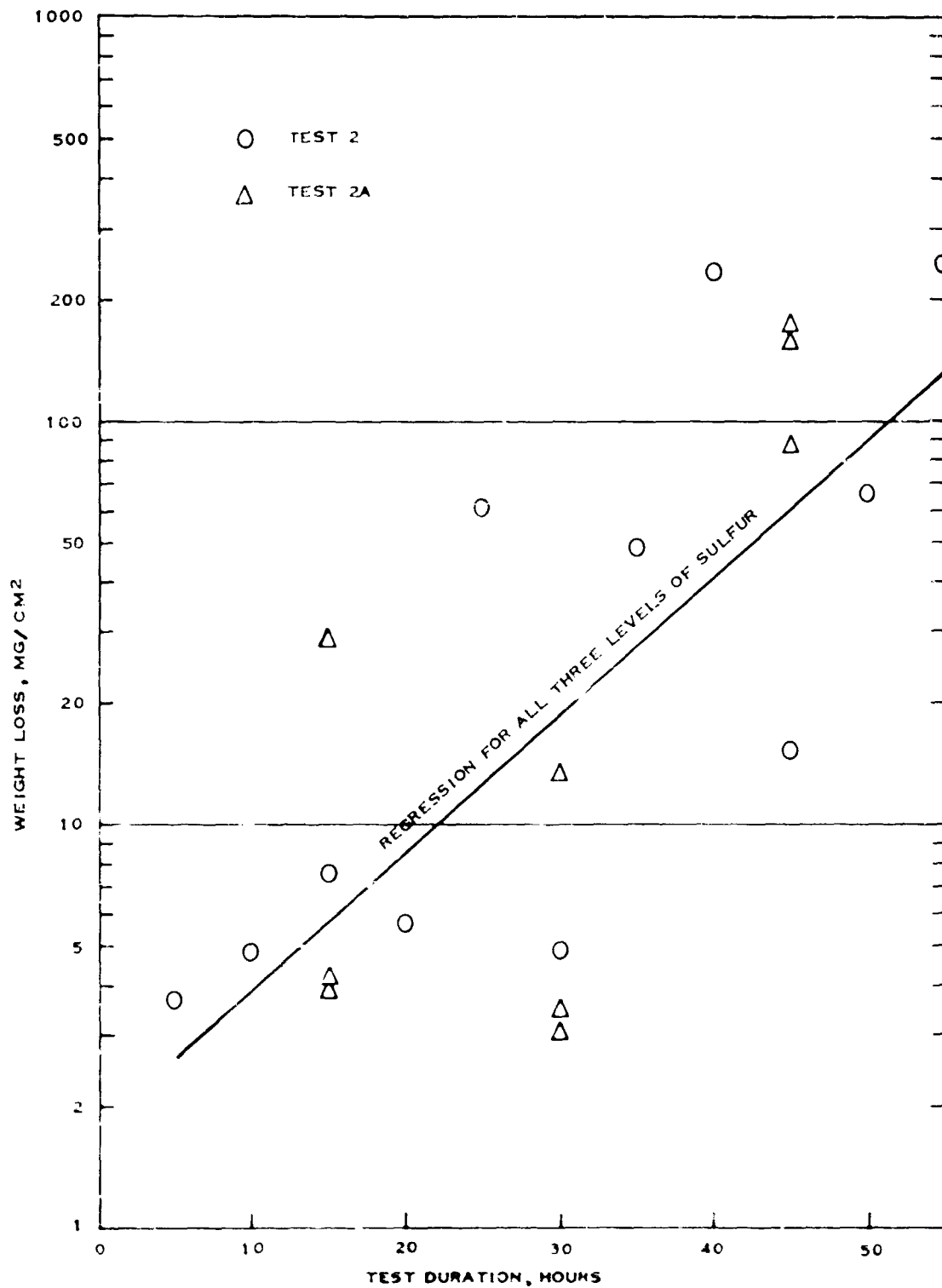


FIGURE 4  
HOT CORROSION OF MISCO MDC-1 COATED MAR M-200 WITH 1.0 PPM  
SEA SALT IN AIR AND <0.0040 WT. % SULFUR IN FUEL

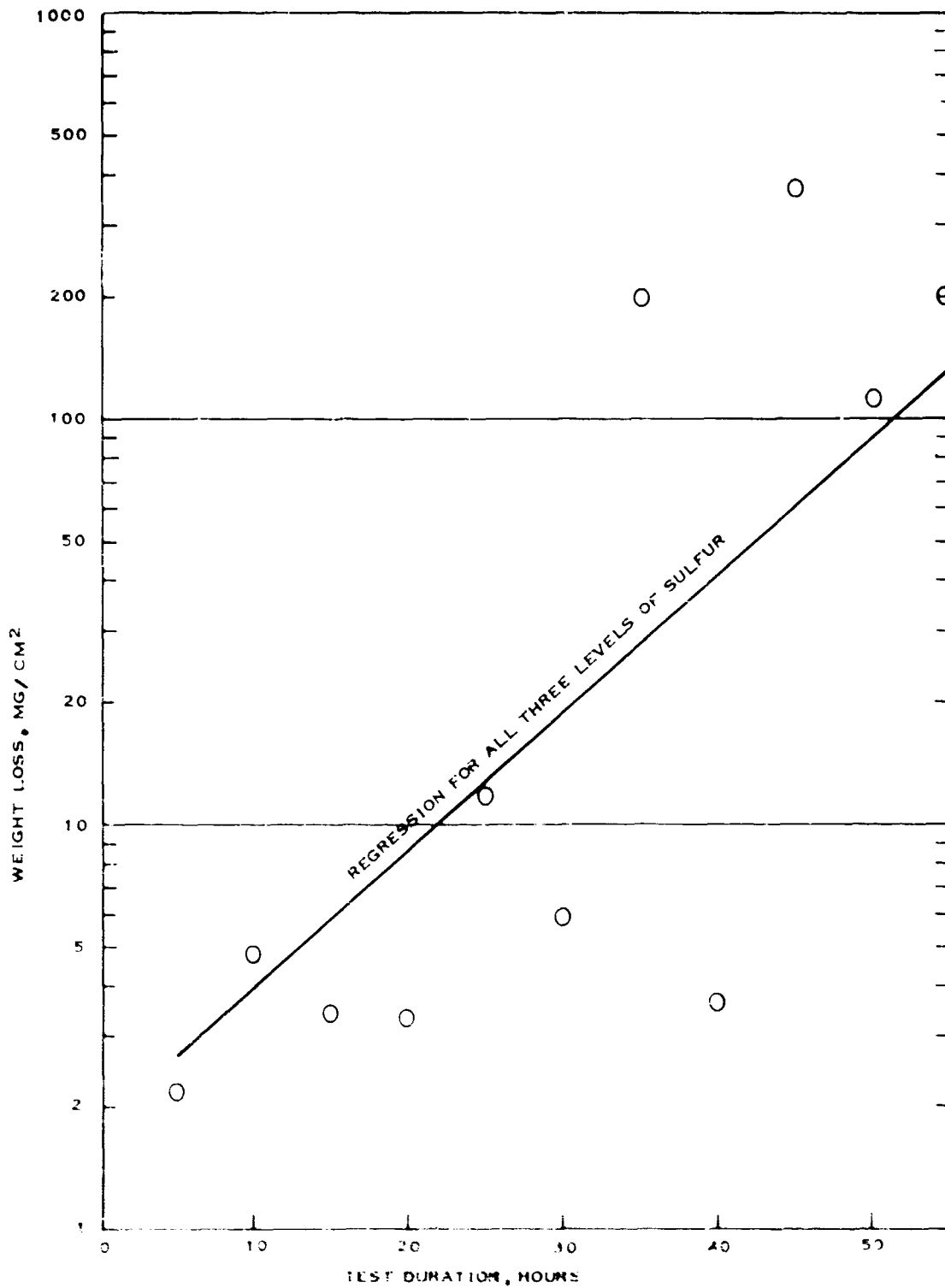


FIGURE 5  
HOT CORROSION OF MISCO MDC-1 COATED MAR M-200 WITH 1.0 PPM  
SEA SALT IN AIR AND 0.040 WT. % SULFUR IN FUEL

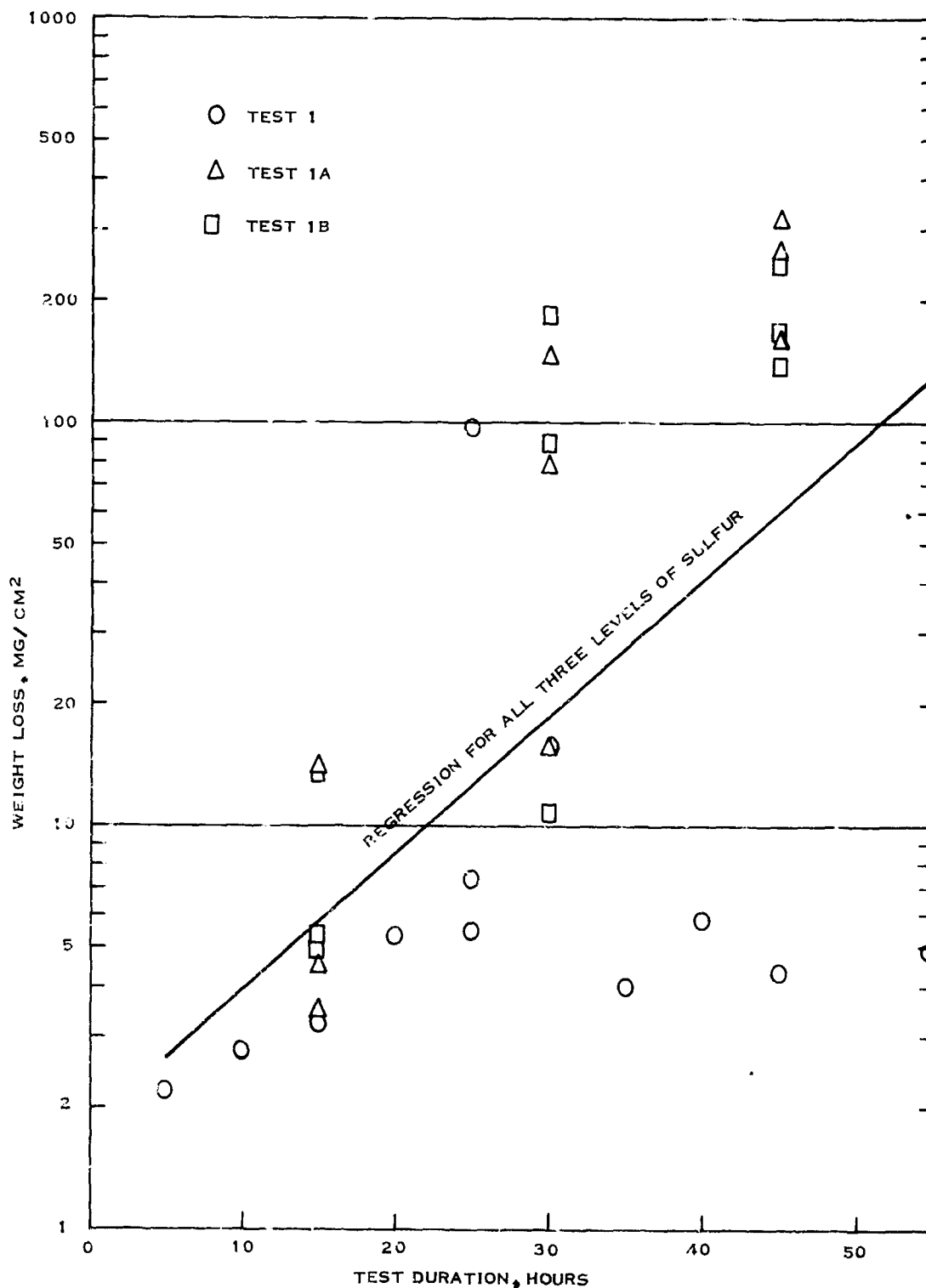


FIGURE 6  
HOT CORROSION OF MISCO MDC-1 COATED MAR M-200 WITH 1.0 PPM  
SEA SALT IN AIR AND 0.40 WT. % SULFUR IN FUEL



in fuel (Tests 1, 2, and 6) were conducted as part of the initial program. It was apparent from inspection of data from Tests 1, 2, and 6 that there was greater variability of weight-loss data than with other coating-alloy systems evaluated previously. Additional tests were conducted with the low and high concentrations of sulfur in fuel ( $<0.0040$  and  $0.40$  weight per cent). In the repeat tests the plan for specimen removal was modified, as shown in Section 10, to provide three specimens at each of three exposure times (15, 30, and 45 hours). Each specimen was exposed for equal time in each of the three stages of the cascade to remove any effect of position in the cascade on metal weight loss. The data from the additional tests also showed a large variability and operating data for the six tests at the three levels of sulfur in fuel with  $1.0$  ppm sea salt were examined carefully for malfunction of equipment, non-specification control of variables or other explanation for the wide spread in weight-loss data. In the absence of any justification for discarding a portion of the data, all specimens are included in the following analysis of the data.

In the second repeat test (Test 1B) with  $0.40$  weight per cent sulfur in fuel and  $1.0$  ppm sea salt in air a reference specimen of bare Inconel 713C was mounted at the rear of the cascade and the specimen was replaced with a new specimen at each 5-hour period. Thus, nine reference specimens were available for evaluation of variability from period-to-period during the test. The weight-loss data for the reference specimens are shown in Table 4. The standard deviation of the weight-loss data for the bare Inconel 713C reference specimens in terms of logarithms was calculated to be  $0.298$  or a percentage standard deviation of 99 per cent of the value. These data compare favorably with the data obtained in Reference 1 ( $0.288$  or 94 per cent) for 5-hour tests with bare alloys and supports the belief that variability of weight-loss data in the current program is not a function of test-rig operation.

The data for each of the three concentrations of sulfur in fuel with  $1.0$  ppm sea salt in air were fit to equations of the form of Equation 3. An Analysis of Covariance was made of these data and is shown in Table 5. This analysis showed no significant difference in the relative rates of corrosion or in the adjusted mean weight losses using a 95 per cent confidence level. Thus, a single regression equation will express all of the data. The lines on Figures 4 to 6 represent a common regression equation.

The standard deviation of the weight-loss data in terms of logarithms was  $0.557$  or a percentage standard deviation of 260 per cent of the value. This standard deviation is much larger than obtained in Reference 1 ( $0.288$ ) or with the high-weight-loss group of data with MDC-1 coated Inconel 713C ( $0.352$ ).

The geometric mean for all weight-loss data with the three concentrations of sulfur in fuel in the presence of  $1.0$  ppm sea salt in air was  $192 \text{ mg/cm}^2$ . This weight loss is nearly two orders-of-magnitude larger than for weight losses of specimens exposed in the absence of sea salt in air. The relative rates of corrosion for the three levels of sulfur in fuel in the

TABLE 4  
WEIGHT-LOSS DATA FOR INCONEL 713C REFERENCE SPECIMENS  
(Test 1B)

<u>Test Period</u>	<u>Specimen Weight Loss</u>		
	<u>mg</u>	<u>mg/cm<sup>2</sup> (a)</u>	<u>log mg/cm<sup>2</sup></u>
1	157.6	14.34	1.156549
2	254.0	23.11	1.363800
3	42.6	3.88	0.588832
4	91.3	8.31	0.919601
5	264.3	24.05	1.381115
6	268.6	24.44	1.388101
7	79.5	7.23	0.859138
8	108.2	9.84	0.992995
9	57.8	5.26	0.720986

Geometric Mean, mg/cm <sup>2</sup>	11.00
Standard Deviation, log mg/cm <sup>2</sup>	0.298054
Coefficient of Variation, %	99

(a) Mg times 0.090991 = mg/cm<sup>2</sup>.

TABLE 5  
ANALYSIS OF COVARIANCE OF MISCO MDC-1 COATED MAR M-200 DATA  
 (1.0 ppm Sea Salt in Air)

Line	df	$\sum x^2$	$\sum xy$	$\sum y^2$	b	Deviation From Regression	
						df	Mean Square
1	<0.0040	19	4	100.00	143.980145	9.134828	0.035117
2	0.040	10	2	750.00	113.757075	7.555103	0.041366
3	0.40	22	5	070.00	160.269733	15.635095	0.031611
4	Within						
5	Reg. Coef.					55	17.496814
6	Common	58	11	920.00	418.006953	2	0.169671
7	Adj. Means					57	17.666485
8	Total	60	11	935.2459	417.845231	2	0.054200
						59	17.720685
							0.318123
							0.084835 (-)
							0.039938
							0.027100 (-)

(-) No significant difference at the 95 per cent confidence level.  
 $\sqrt{C.309938} = 0.556723$  (260 per cent) = Standard Deviation

Log Y = 0.035068 X + 0.213798      Equation using common regression coefficient.  
 where: Y = weight-loss, mg/cm<sup>2</sup>  
         X = exposure time, hours

presence of 1.0 ppm sea salt in air (0.035068) is nearly an order of magnitude larger than for the data in the absence of sea salt in air (0.004127). From these data it can be concluded that sea salt in air is the principal causative agent for hot corrosion of Misco MDC-1 coated Mar M-200 specimens under these conditions.

#### 4.3 Metallography

Cross-section coupons from selected specimens of Misco MDC-1 coated Mar M-200 were examined; as detailed in Appendix 1, Section 8.5. of this report. The uniformity and thickness of the aluminum-diffusion coating was appraised by metallographic examination of several specimens, as received from Misco. Similarly, the mode and intensity of corrosive attack was evaluated following exposure over the range in conditions of this investigation.

In particular, evidence was sought concerning the suspected lack of uniformity in the coating on unexposed specimens, discussed previously. Also, the absence of deep-intercrystalline corrosion must be established for the valid use of loss in weight by the specimen to measure metal damage, as in the preceding section.

A metallographic cross-section of a satisfactory MDC-1 coated Mar M-200 specimen as received from Misco is shown in Figure 7. The aluminum-type coating has a total depth of approximately 2 mils, which is divided about equally between an outer-layer with non-metallic dispersions and a diffusion-layer. Unfortunately, the coating thickness varied from approximately 0.5 to 2.0 mils on the unexposed specimens examined. A typical example of defective coating is shown in Figure 8. This variability in coating thickness could easily account for the relatively large experimental error found with this coating-alloy system, which was discussed in the preceding section.

It is not known whether the variability in coating thickness is a characteristic of the coating-alloy system, or the specific heat of Mar M-200, or the specific pack of MDC-1. Consultation with Misco revealed that their experience with the application of MDC-1 coating to Mar M-200 was very limited; however, they felt that the variability in coating thickness was not a characteristic of the coating-alloy system. Also, they did not think that it could be attributed to segregation in alloy composition during casting. Rather, they felt that it probably was the result of uneven heating of the pack during the vapor-coating process. This suspicion is based upon the use of a small-research furnace for coating our specimens.

Field experience by the Navy has indicated that variability in coating thickness on turbine blades is not unusual, and is a production problem. In this light, the data obtained from our investigation with this coating-alloy system has been treated as a valid sample.

Previous experience (2) has shown that the appearance of MDC-1 coating is not altered by the electro-cleaning procedure used to remove



METALLOGRAPHIC CROSS-SECTION. 3% SULFURIC ACID-ELECTROLYTIC ETCHED.  
2000X MAGNIFICATION.

FIGURE 7  
UNEXPOSED MISCO MDC-1 COATED MAR M-200 SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF UNEXPOSED SPECIMEN,  
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

FIGURE 8  
DEFECTIVE MISCO MDC-1 COATING ON MAR M-200 SPECIMEN

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deposits and scale following exposure. This was confirmed for MDC-1 coated Mar M-200 by metallographic examination of cross-section coupons from several specimens which had been electro-cleaned.

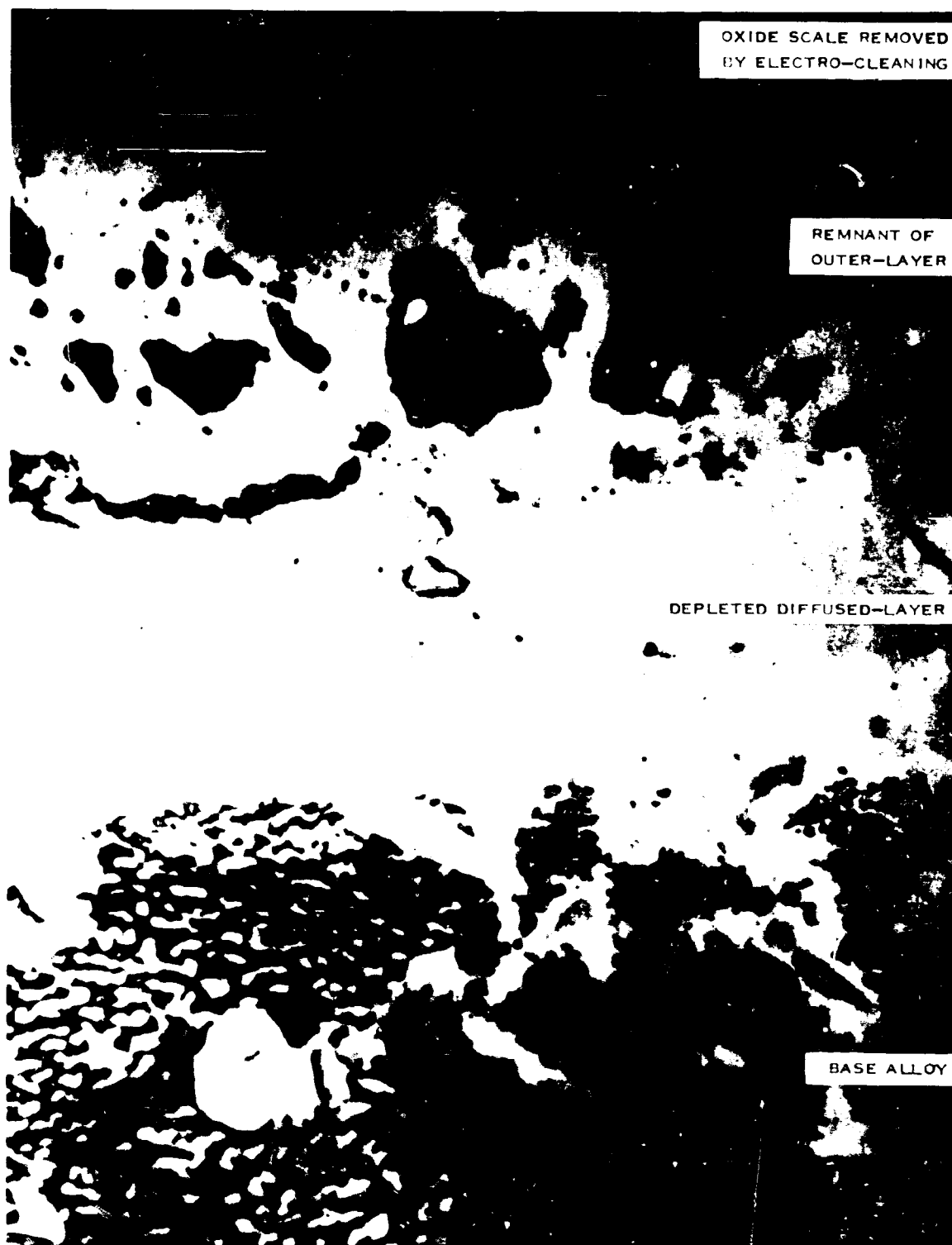
In those areas where the MDC-1 coating remained intact following exposure, evidence was found of gradual diffusion of the aluminum into the base alloy. This depletes the diffused-layer, and increases its depth, as shown in Figure 9. This modification of the coating remnant became increasingly evident with increasing exposure time, but was not favored by any other exposure variable in this program.

Exposure of Misco MDC-1 coated Mar M-200 in an environment free of sea salt resulted in only slight, uniform, surface oxidation. In some areas oxide penetration was observed to have progressed into the depleted diffused-layer of the coating, as shown in Figure 10. A few instances of complete coating failure were observed, as shown in Figure 11, with surface oxidation preceded by a limited zone of alloy depletion. It should be noted that in this environment (2000 F) with the high-sulfur fuel (0.40 wt %) no evidence was found of sulfide penetration in either the depleted diffused-layer of the coating (Figure 10) or the depleted alloy (Figure 11), despite oxide penetration.

With the addition of sea salt, via the air, to this environment, characteristic hot corrosion was encountered. Typical attack on the MDC-1 coating is shown in Figure 12; with separation of the outer-layer from the diffused-layer by corrosion products, which frequently resulted in loss of the outer-layer of the coating. Also, penetration of the diffused-layer of the coating by randomly-dispersed, light-grey, globules of metallic sulfides was usual, and is evident in Figure 12. The addition of sulfur, via the fuel, to this environment did not perceptively alter the attack.

In those areas where the MDC-1 coating was penetrated by hot corrosion, the mode and intensity of attack was similar to that observed on the bare alloy (1). In general, the attack was less severe on the rear-face of the specimens, which is shown in Figure 13. The accelerated-oxidation attack, that is typical of hot corrosion, was evident on the impact-face of specimens, which is shown in Figure 14. It should be noted that, characteristically, the attack advances on a broad front without deep-intercrystalline penetration of sulfides or oxides.

Limited evidence of intercrystalline oxidation, to a depth of up to 8 mils, was found in a few specimens at the point of flexure from support by the holder, as shown in Figure 15. This photomicrograph is presented to illustrate the type of attack which would vitiate the use of loss in weight by the specimen as a measure of metal damage. However, such intercrystalline corrosion was not found to be extensive enough under the conditions of our investigation to be of practical importance.



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 50 HOURS AT 2000 F TEST CONDITION  
WITH NO SEA SALT IN AIR AND  $<0.0040$  WT % SULFUR IN FUEL.  
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

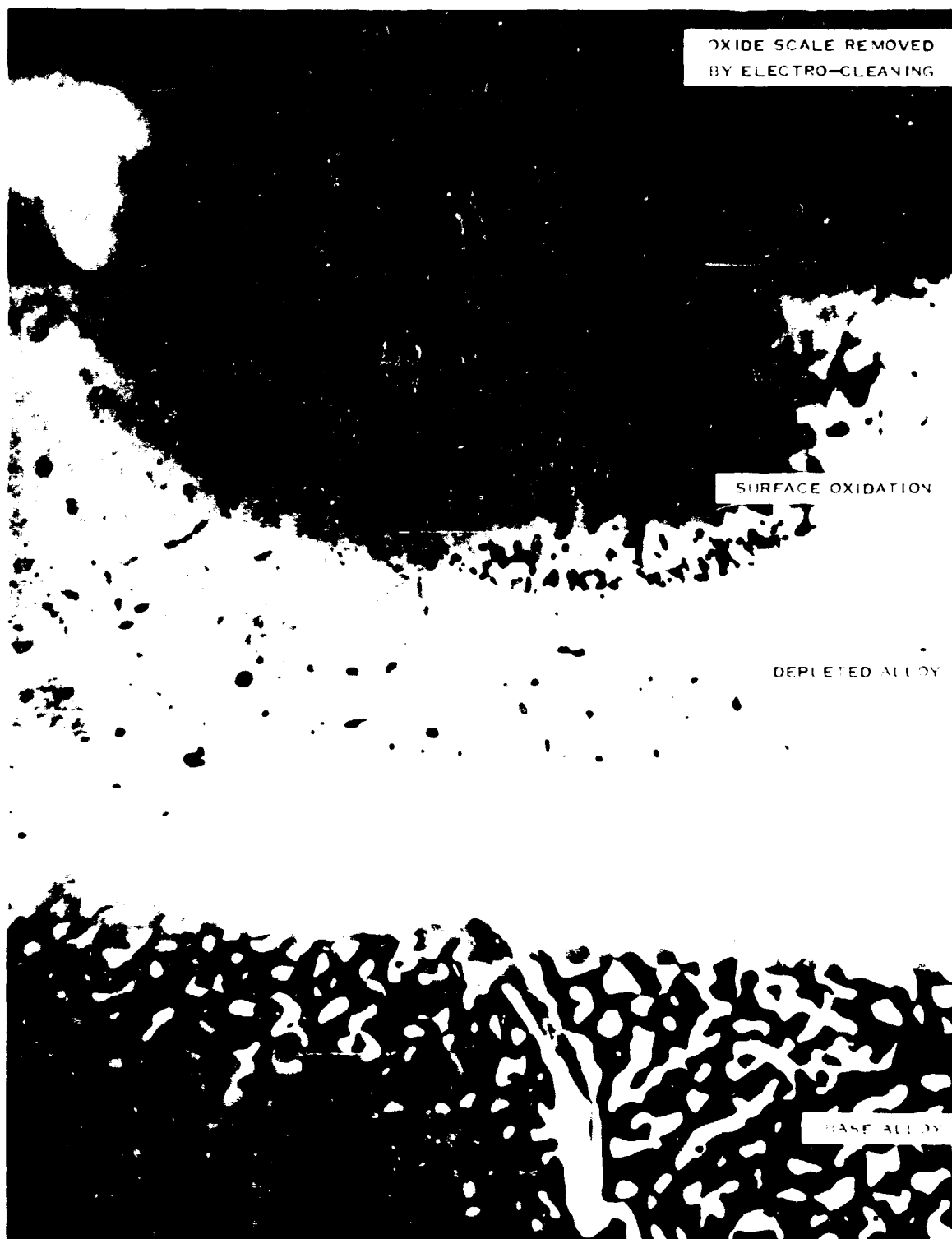
FIGURE 9  
DEPLETION OF MISCO MDC-1 COATING ON MAR M-200 SPECIMEN





METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 2000 F TEST CONDITION  
WITH NO SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.  
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

FIGURE 10  
OXIDATION OF MISCO MDC-1 COATING ON MAR M-200 SPECIMEN



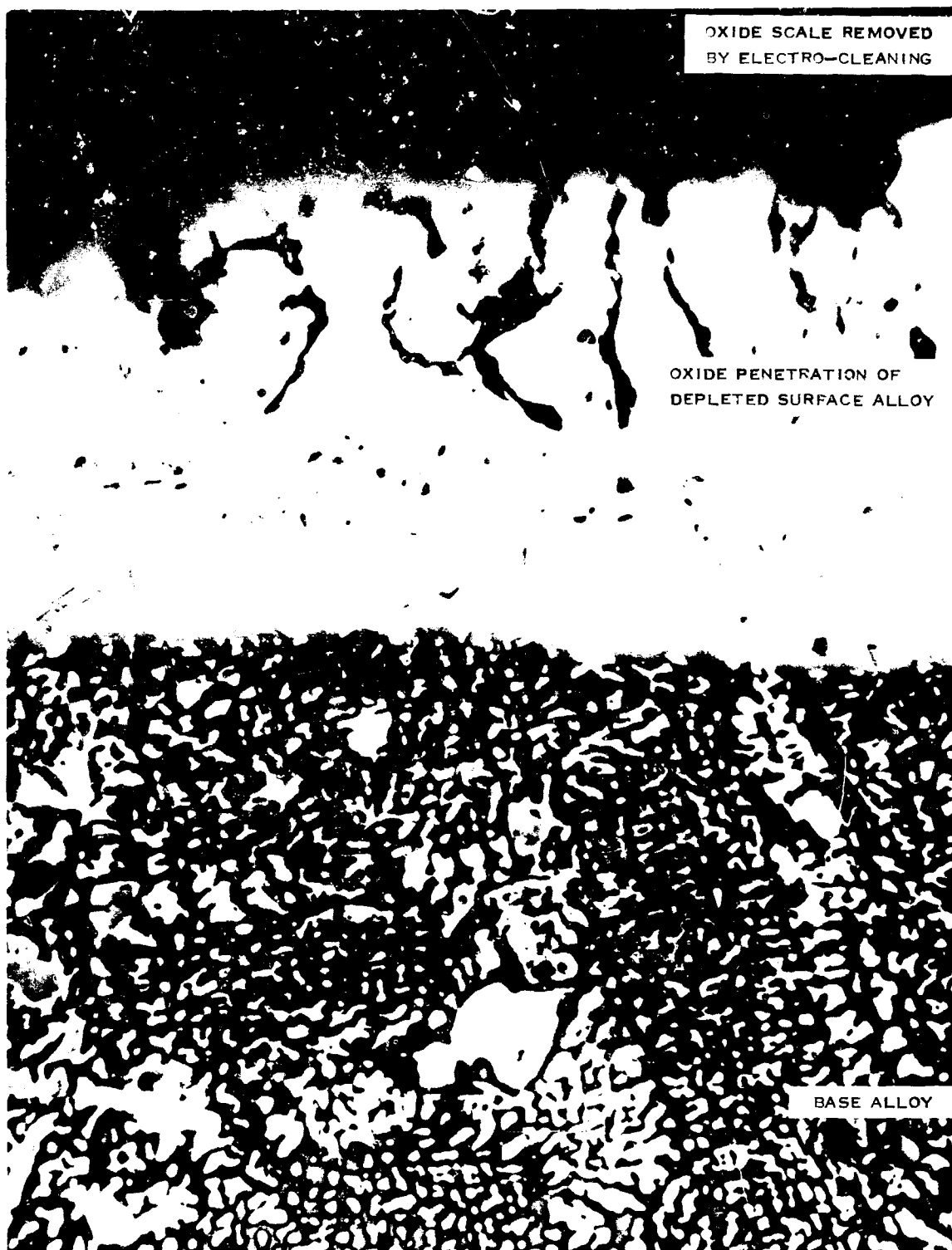
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 50 HOURS AT 2000 F TEST CONDITION  
WITH NO SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.  
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

FIGURE 11  
OXIDATION OF MISCO MDC-1 COATED MAR M-200 SPECIMEN



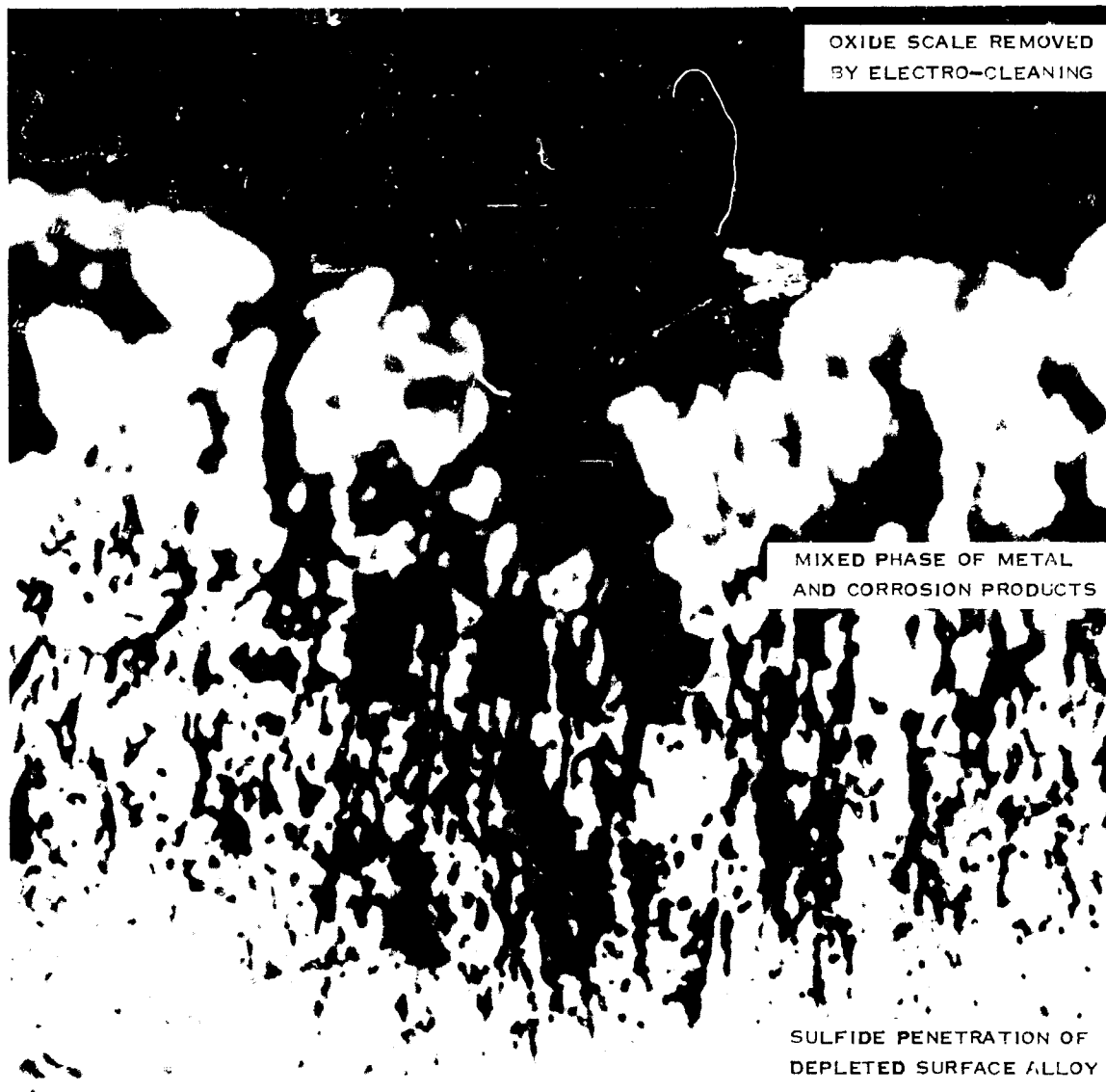
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 50 HOURS AT 2000 F TEST CONDITION  
WITH 1 PPM SEA SALT IN AIR AND  $<0.0040$  WT % SULFUR IN FUEL,  
3% SULFURIC ACID-ELECTROLYTIC ETCHED, 2000X MAGNIFICATION.

FIGURE 12  
OXIDATION OF MISCO NDC-1 COATING ON MAR M-200 SPECIMEN



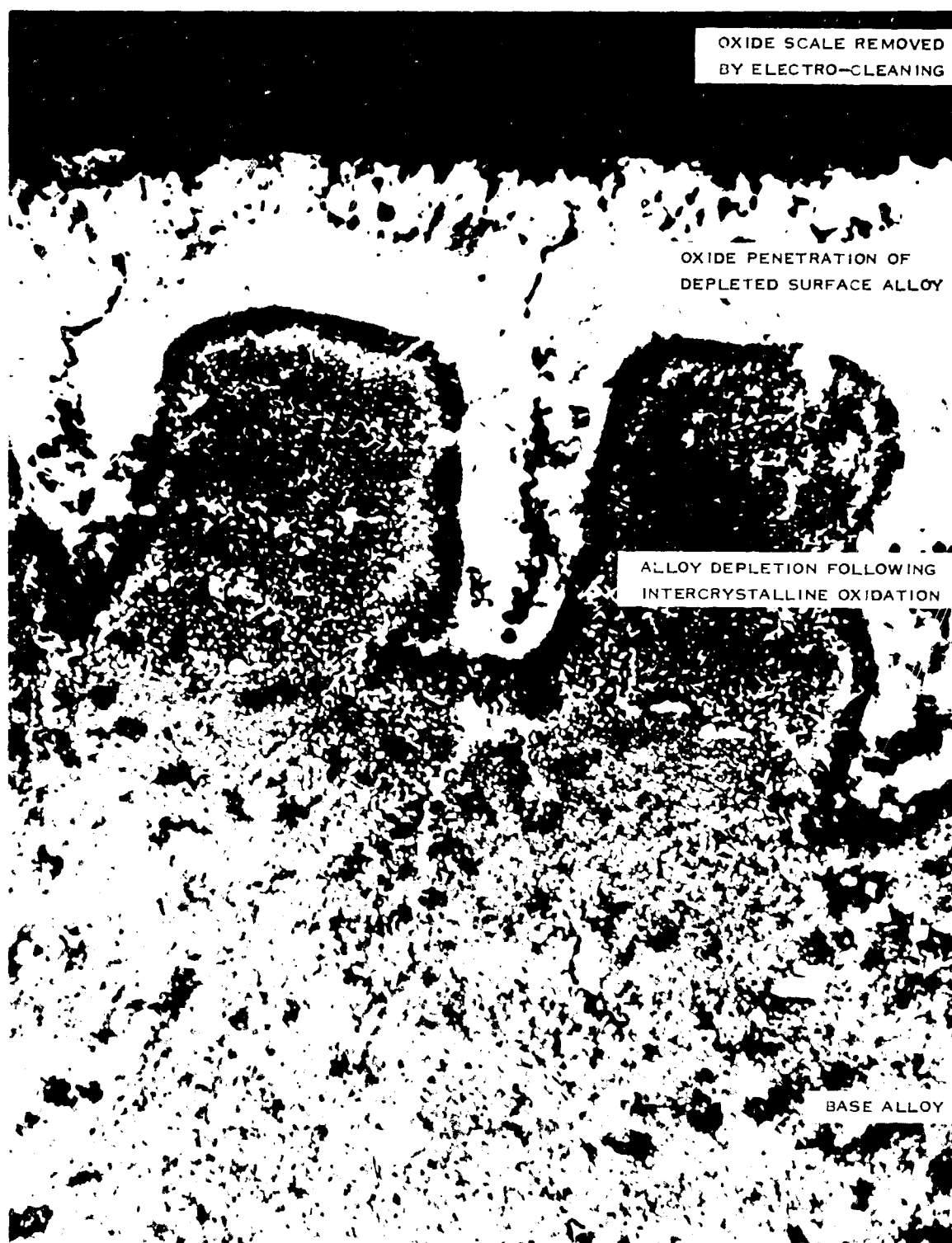
METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 2000 F TEST CONDITION  
WITH 1 PPM SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL.  
3% SULFUR ACID-ELECTROLYTIC ETCHED. 2000X MAGNIFICATION.

FIGURE 13  
OXIDATION OF MISCO MDC-1 COATED MAR M-200 SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 25 HOURS AT 2000 F TEST CONDITION  
WITH 1 PPM SEA SALT IN AIR AND 0.40 WT % SULFUR IN FUEL,  
3000X MAGNIFICATION.

FIGURE 14  
ACCELERATED OXIDATION OF MISCO MDC-1 COATED MAR M-200 SPECIMEN



METALLOGRAPHIC CROSS-SECTION OF SPECIMEN AFTER 45 HOURS AT 2000 F TEST CONDITION  
WITH 1 PPM SEA SALT IN AIR AND 0.4 WT % SULFUR IN FUEL.  
3% SULFURIC ACID-ELECTROLYTIC ETCHED. 500X MAGNIFICATION.

FIGURE 15  
INTERCRYSTALLINE OXIDATION OF MISCO MDC-1 COATED MAR M-200 SPECIMEN

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Several specimens which had been exposed under comparable conditions, but differed greatly in weight loss, were examined. No basic differences in either the coating or base alloy were observed which would explain their response.

Normally, the corrosive attack experienced by the Misco MDC-1 coated Mar M-200 specimens during this investigation resulted in only shallow penetration of corrosion products, despite the catastrophic rates of metal loss encountered in the presence of sea salt. The depth of surface penetration was usually less than one mil. This serves to justify the use of the metal-loss data as a valid measurement of the extent of corrosive attack on this coating-alloy system over the range of conditions investigated.

## 5. FUTURE WORK

The effect of sulfur in fuel was found to vary among the various bare superalloys used in previous programs (1); i.e., a reduction in fuel sulfur had no effect with some superalloys, and increased hot corrosion with two superalloys under some conditions. With three coating-alloy systems in Reference 2 and the current program the effect of sulfur in fuel was found to increase, decrease or have no effect on hot corrosion.

In previous programs (1) it was found that the effect of sulfur in fuel on hot corrosion varied with temperature. Statistically significant decreases in hot corrosion were found with reductions in fuel sulfur at temperatures below the melting point of sodium sulfate (1623 F) with superalloys that were unaffected by changes in sulfur content at higher temperatures.

To complete the investigation of the effect of sulfur in fuel on hot corrosion of bare and coated superalloys in a marine environment a number of coating-alloy systems should be evaluated at one or more temperatures below the melting point of sodium sulfate. With coated superalloys at the 2000 F test condition it has been necessary to increase exposure time up to 55 hours to obtain sufficient attack of the test specimens for evaluation. With coating-alloy systems the variability of weight losses in the presence of sea salt in air has been large and duplicate tests have been required for evaluation. It is estimated that at lower temperatures the time of exposure may have to be extended further to provide weight losses for evaluation.

With the number of evaluations required to complete the program and the time required for each test, productivity with the current six-position test rig is becoming a problem. To expedite testing for the remainder of the program a new test section is being designed for the hot corrosion rig. The new design will maintain the same capability for operating at high pressure, high temperature, high velocity and realistic stoichiometry as available with the present test rig. It is anticipated that 48 test specimens will be arranged in a rotating fixture in the gas stream in such a manner that no specimen will be in the shadow of another specimen and the specimens will thus receive uniform exposure. With the revised test fixture it is proposed to use round test pins as specimens rather than the flat bars used in previous programs. This should facilitate the use of depth of penetration as well as weight loss as an evaluation criteria. With the number of specimen locations available, reference specimens could be used to monitor any variability of test rig operation during the extended tests.



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Statistical Analysis by M. R. Goss and Lynn Jones.

Metallographic Analysis by E. H. Borgman and Velma Gooch.

7. REFERENCES

1. Schirmer, R. M. and Quigg, H. T., "Effect of JP-5 Sulfur Content on Hot Corrosion of Superalloys in Marine Environment". Progress Report No. 3 for Naval Air Systems Command Contract NOw 65-0310-d. Phillips Petroleum Company Research Division Report 4370-66R. Bartlesville, Oklahoma, July 1966, 173 pp.
2. Quigg, H. T. and Schirmer, R. M., "Effect of Sulfur in JP-5 Fuel on Hot Corrosion of Coated Superalloys in Marine Environment". Progress Report No. 3 for Naval Air Systems Command Contract NOw 66-0263-d. Phillips Petroleum Company Research Division Report 4706-67R. Bartlesville, Oklahoma, April 1967, 111 pp.
3. Whitfield, M. G. and Parzuchowski, R. S., "Sulfidation Resistant Coatings". Preprint of Paper No. 115 presented at the Sixty-Ninth Annual Meeting of ASTM in Atlantic City, N. J., June 27 - July 1, 1966.
4. Danek, G. J., "State-of-the-Art Survey on Hot Corrosion in Marine Gas-Turbine Engines". U. S. Navy Marine Engineering Laboratory Report 32/65. Annapolis, Md. March 1965, 33 pp.
5. Snedecor, George W., "Statistical Methods". Fifth Edition. Published by The Iowa State University Press, Ames, Iowa. 1962.
6. Fromm, E. H., "Design and Calibration of the Improved Phillips Fuel Testing Facilities". Phillips Petroleum Company Research Division Report 3527-63R. Bartlesville, Oklahoma, July 2, 1963.
7. Shirley, H. T., "Effects of Sulphate-Chloride Mixtures in Fuel-Ash Corrosion of Steels and High-Nickel Alloys". Journal of the Iron and Steel Institute, Vol. 82, February 1956, pp. 144-153.
8. Blade, O. C., "Aviation Turbine Fuels, 1966". Petroleum Products Survey No. 49. Bartlesville, Oklahoma, Petroleum Research Center, Bureau of Mines, U. S. Department of the Interior, March 1967.
9. Fromm, E. H., Quigg, H. T. and Schirmer, R. M., "Effect of JP-5 Sulfur Content on Hot Corrosion of Superalloys in Marine Environment". Progress Report No. 2 for Bureau of Naval Weapons Contract NOw 65-0310-d. Phillips Petroleum Company Research Division Report 4319-66R. Bartlesville, Oklahoma, January 31, 1966, 62 pp.
10. Hall, N. A., "Mean Specific Heats for the Working Media of Gas Turbine Powerplants". SAE Quarterly Transactions, Vol. 1 No. 3, pp 490-497, July 1947.

8. APPENDIX 1  
(Test Equipment)

8.1. Test Facility

Phillips research facility for testing jet fuel, pictured in part in Figure 16 has been described previously in detail by Fromm (6). Air is supplied by rotary Fuller compressors and filtered by a Selas Vape-Sorber. This air is preheated just before it enters the 2-inch combustor testway by a Thermal Research heat-exchanger. Both fuel and sea water are supplied by nitrogen pressurization of their respective tanks. A portion of the metering and automatic control equipment can be seen in Figure 17. Air flow rates up to 2.0 lb/sec, at inlet air pressures up to 15 atmospheres, and inlet air temperatures up to 1400 F are attainable.

8.2. Phillips 2-Inch Combustor

A scale diagram of the 2-inch combustor used in this study is shown in Figure 18. Design details of the combustor are presented in Table 6. Basically, it embodied the principal features of combustors used in modern aircraft-turbine engines. It was a straight-through can-type, combustor with fuel atomization by a single, simplex-type, nozzle. The combustor liner was fabricated from 2-inch, Schedule 40, Inconel pipe, with added internal deflector skirts for film cooling of surfaces exposed to the flame.

A scale diagram of the Phillips test rig used in this study of hot corrosion is shown in Figure 19. Its design permits easy access to the fuel nozzle, combustor liner, test specimens, etc. The combustor installation was disassembled, inspected, and reconditioned after every test period.

Four chromel-alumel thermocouples were mounted on equal area centers at the location indicated in Figure 19, for measurement of exhaust gas temperature. The thermocouples were housed in  $\frac{1}{4}$ -inch diameter Inconel sheaths for protection.

The sea water injection point was located in the quench zone of the combustor, as indicated in Figure 19, rather than upstream of the combustor or in the primary-combustion zone. This avoided a severe corrosion problem with the combustor liner, and also insured exposure of test specimens to the desired sea salt concentration. The sea water was divided into two metered portions and introduced through opposing jets to obtain uniform distribution of sea water in air by impingement of the jet streams.

The exhaust section was water jacketed to obtain the desired durability of operation with high-temperature gases.

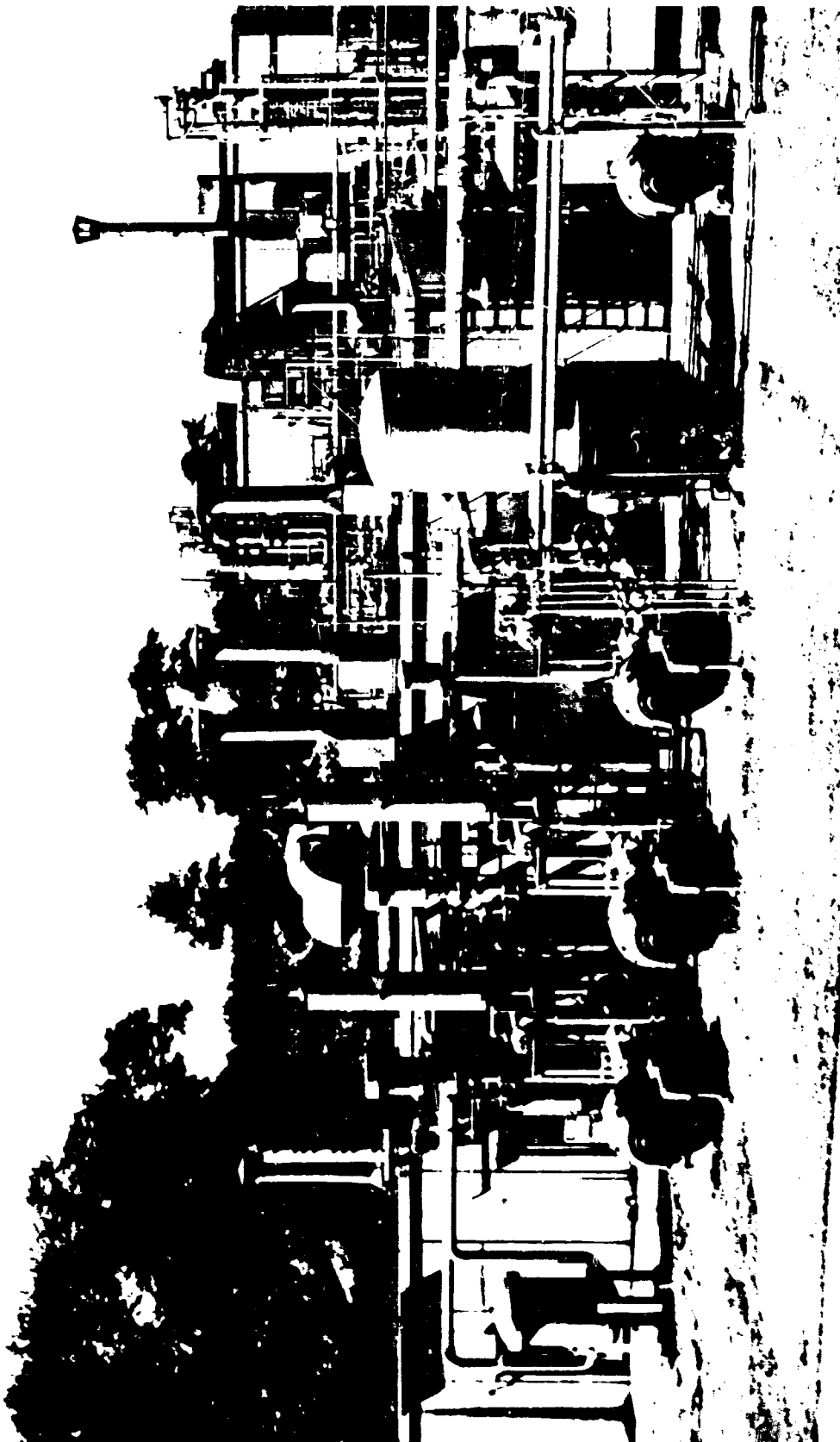


FIGURE 16  
PHILLIPS RESEARCH FACILITY FOR JP FUELS

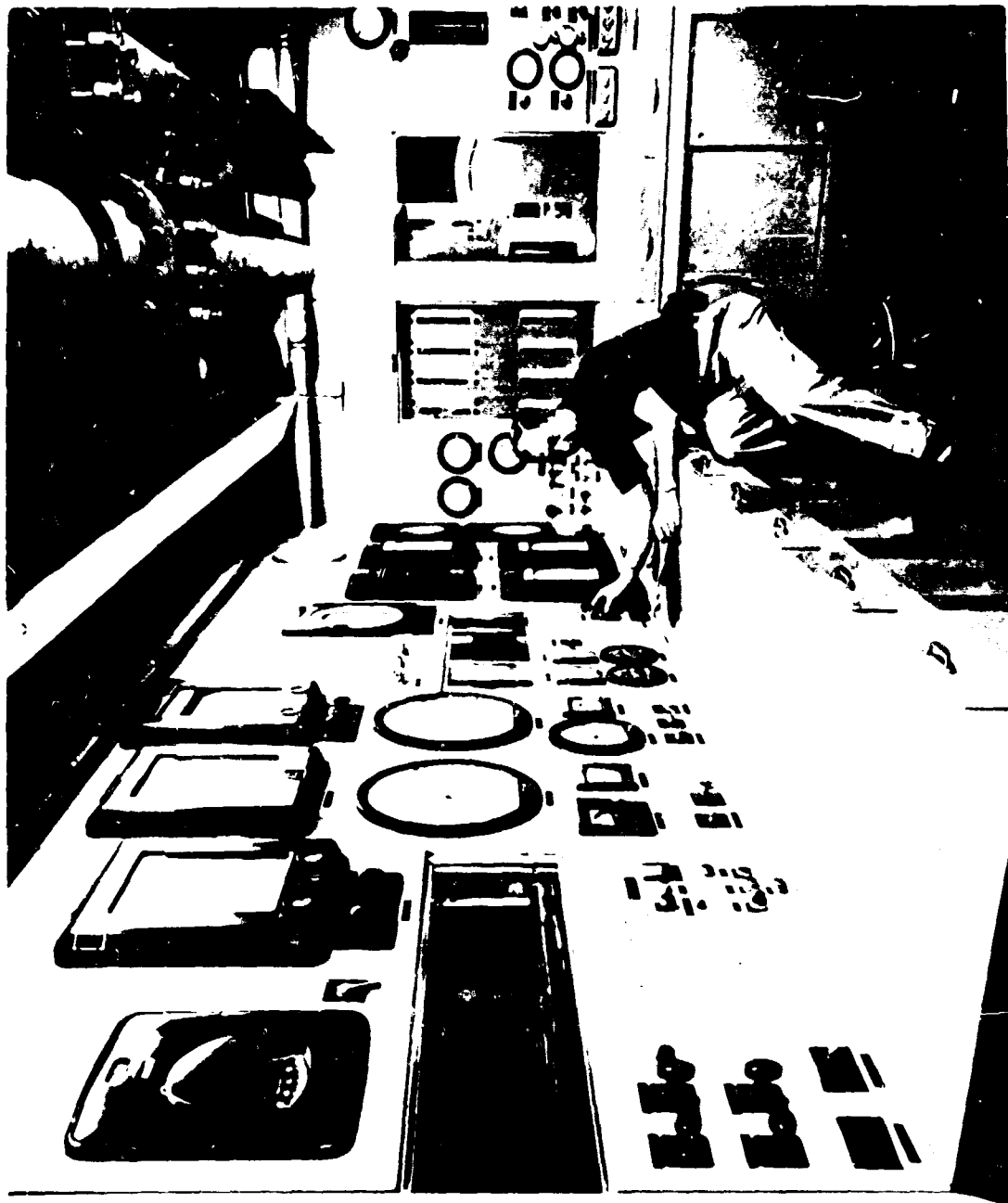
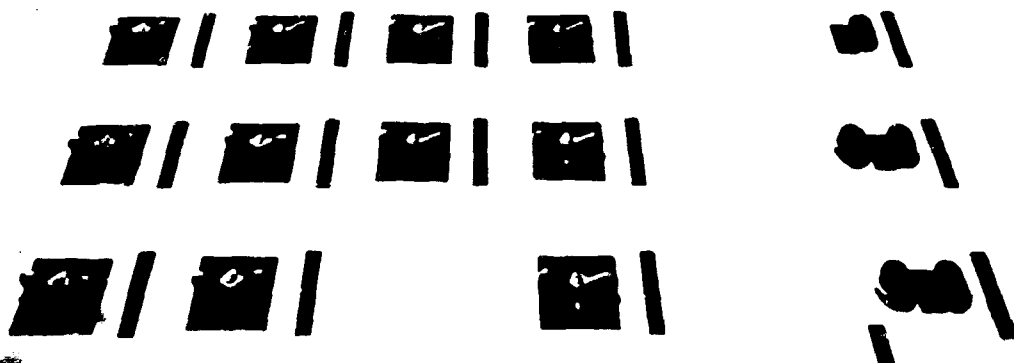


FIGURE 17  
CONTROL ROOM FOR HIGH-PRESSURE COMBUSTOR



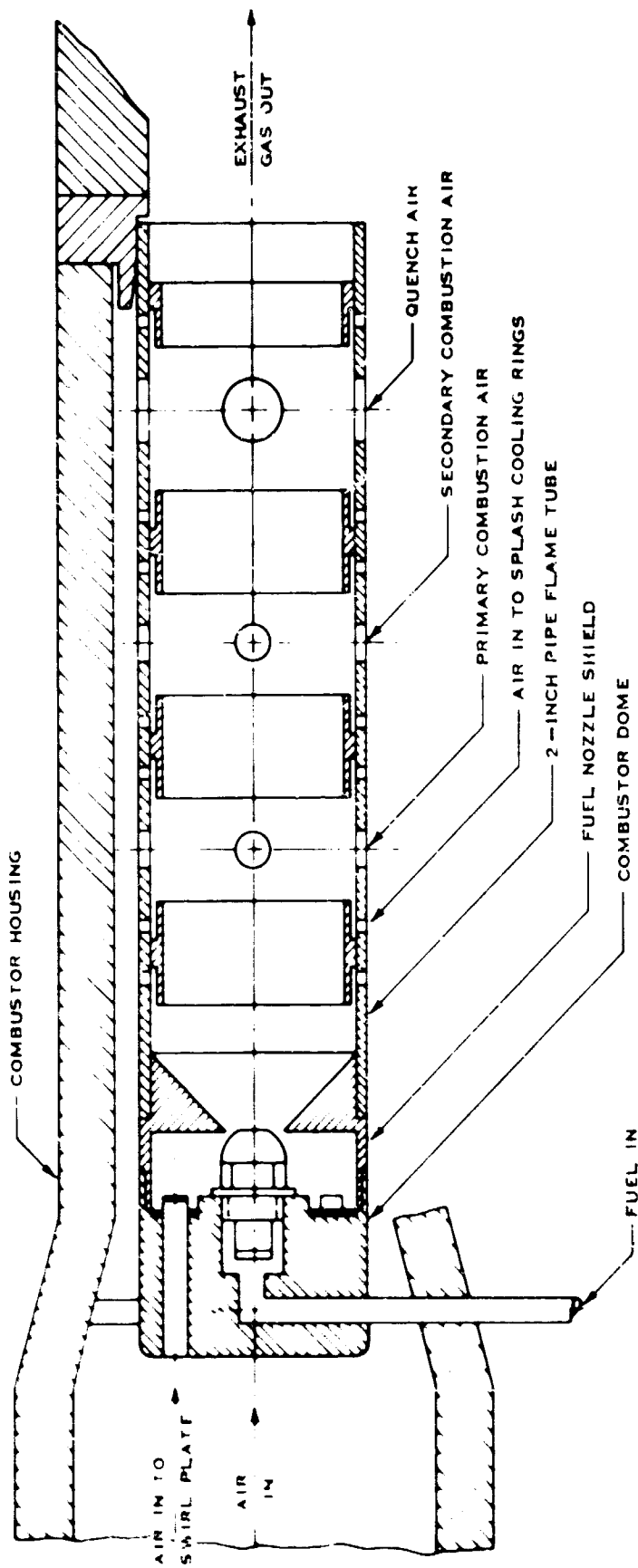


FIGURE 18  
PHILLIPS 2 - INCH COMBUSTOR

TABLE 6

DESIGN DETAILS OF PHILLIPS 2-INCH COMBUSTOR

Combustor Configuration Number	15
Fuel Nozzle	
Type	Simplex (Monarch)
Spray Pattern	Semi-Solid Cone (PLP)
Spray Angle, degrees	50
Capacity, gph of No. 2 Fuel Oil @ 100 psi	13.8
Combustor Dome	
Air Inlet Type	Tangential Swirl
Shield Hole Diameter, in.	0.625
Total Hole Area, sq. in.	0.307
% Total Combustor Hole Area	8.7
Splash Cooling Air	
Hole Diameter, in.	0.125
Holes/Station	16
Number of Stations	7
Total Number of Holes	112
Total Hole Area, sq. in.	1.374
% Total Combustor Hole Area	38.7
Primary Combustion Air	
Hole Diameter, in.	0.250
Total Number of Holes	4
Total Hole Area, sq. in.	0.196
% Total Combustor Hole Area	5.5
Secondary Combustion Air	
Hole Diameter, in.	0.375
Total Number of Holes	4
Total Hole Area, sq. in.	0.442
% Total Combustor Hole Area	12.5
Quench Air	
Hole Diameter, in.	0.625
Total Number of Holes	4
Total Hole Area, sq. in.	1.227
% Total Combustor Hole Area	34.6
Total Combustor Hole Area, sq. in.	3.546
% Cross Sectional Area	133.4

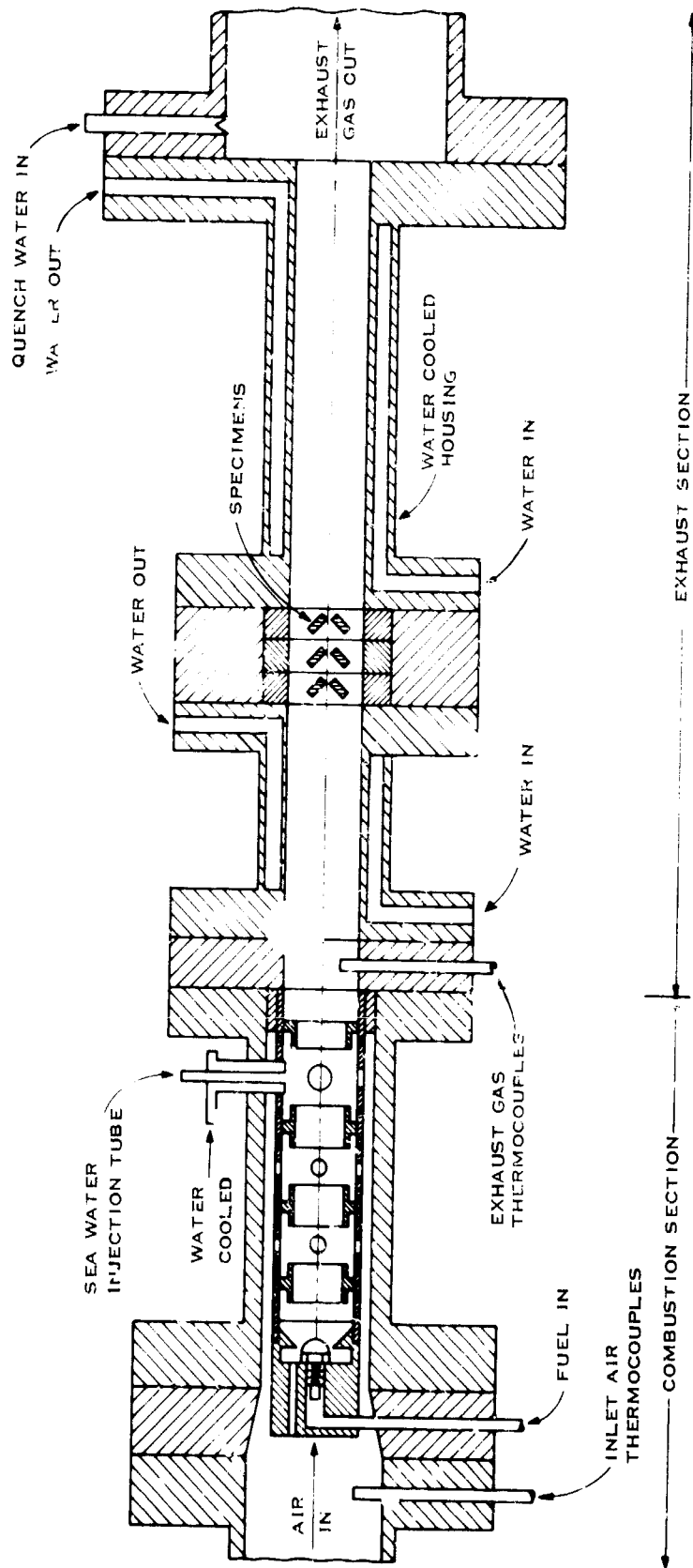


FIGURE 19  
TEST-WAY FOR HOT-CORROSION STUDY



### 8.3. Specimen Holders

The general location of the specimen holders in the exhaust gas from the 2-inch combustor is shown in Figure 19. They are separated from the 2-inch combustor by a 6-inch, water-cooled, spool; and are followed by another 12-inch, water-cooled, spool prior to quench-water injection. Each holder accommodates two metal strips,  $1/8$  by  $1/2$  by  $2\ 3/8$  inches, as shown in Figure 20. Three holders were combined in a cascade, as shown in Figure 21, with each successive holder rotated 120 degrees to prevent channeling of the hot-gas flow. A picture of the specimens mounted in the cascade is shown in Figure 22.

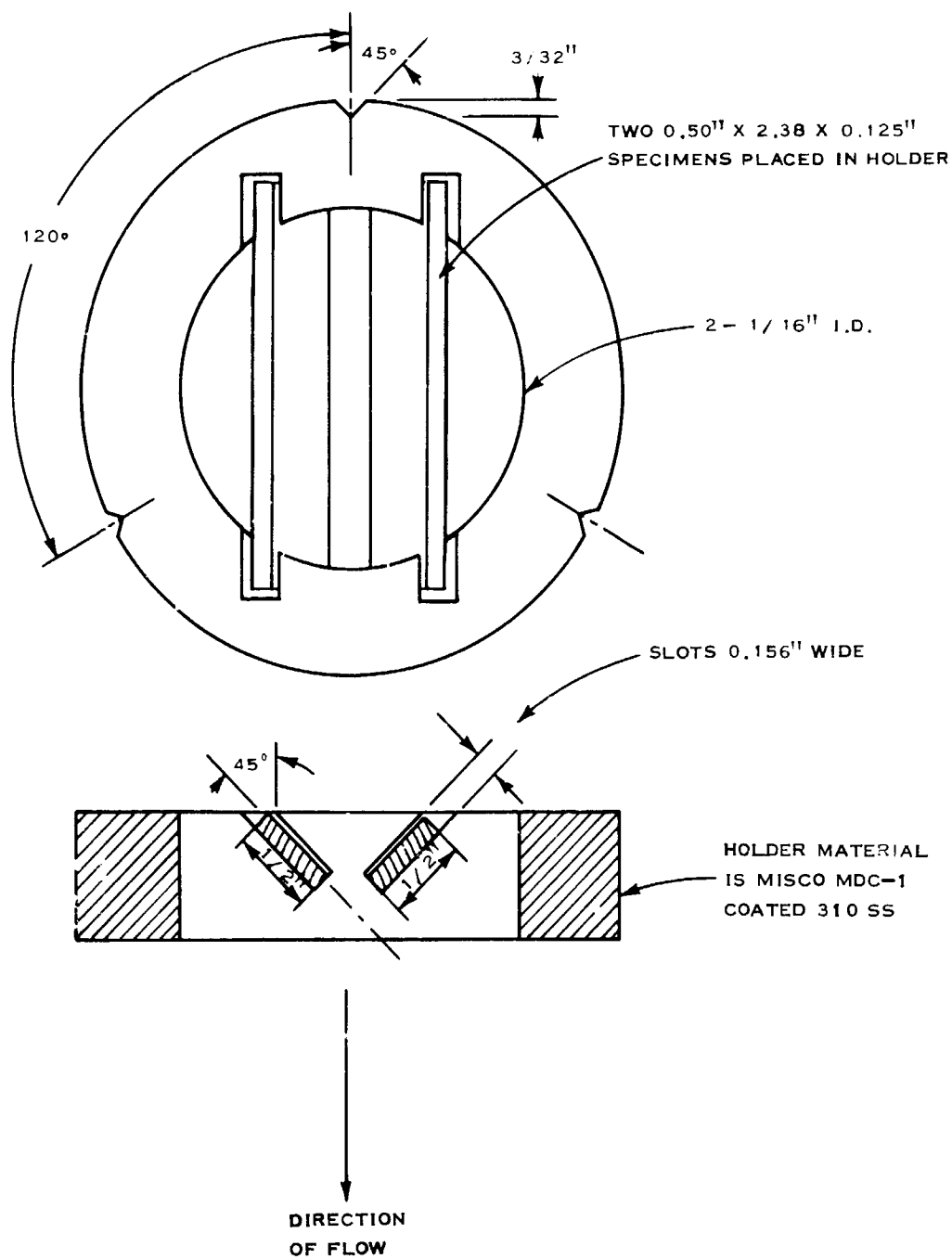
The cross-sectional area of the 2-inch pipe in which the specimen holders are located is 3.36 square inches; however, the unblocked area in the specimen holder is only 1.59 square inches. The holders maintain the specimens at an angle of 45 degrees to the axis of the pipe in which they are located. This provides for acceleration of the gas flow over the surface of the specimens, much as over the turbine blading in an actual engine. The specimens are subjected to appreciable gas-pressure loading while the test is in progress. It is sufficient to slightly bow Inconel 713C specimens at the 2000 F test condition, and the effect increases with exposure time.

### 8.4. Specimen Cleaning

New specimens were cleaned by vapor degreasing with trichloroethylene, using the apparatus shown diagrammatically in Figure 23. Subsequently the specimens were handled with degreased stainless-steel tongs. The initial weight of each test specimen was determined following degreasing.

Following exposure cleaning was necessary to remove the accumulation of surface deposit or scale and to allow for the measurement of metal loss by the specimen from hot corrosion. An electro-cleaning technique was used which has been described by Shirley (7). Briefly, the electro-cleaning technique consists of immersing the specimens in molten sodium hydroxide at 750 to 790 F with  $1/3$  amp/cm<sup>2</sup> passing through the specimen for a period of 10 minutes. The specimens were scrubbed with a wire brush during a water quench, rinsed in acetone, dried, and reweighed. The apparatus used for electro-cleaning is shown diagrammatically in Figure 24. Provisions were made to permit electro-cleaning, simultaneously, of six specimens.

Ten minutes of electro-cleaning, which in previous studies was sufficient to remove all visible scale, did not remove all of the surface scale or deposits from the specimens of this coating-alloy system and each specimen was cleaned twice to give a clean surface. For reference each of six new unexposed Misco MDC-1 coated Mar M-200 test specimens were cleaned twice and the geometric mean weight loss for the six specimens was 1.61 mg/cm<sup>2</sup> with a coefficient of variation of 30 per cent. This level of



**FIGURE 20**  
**SPECIMEN HOLDER**

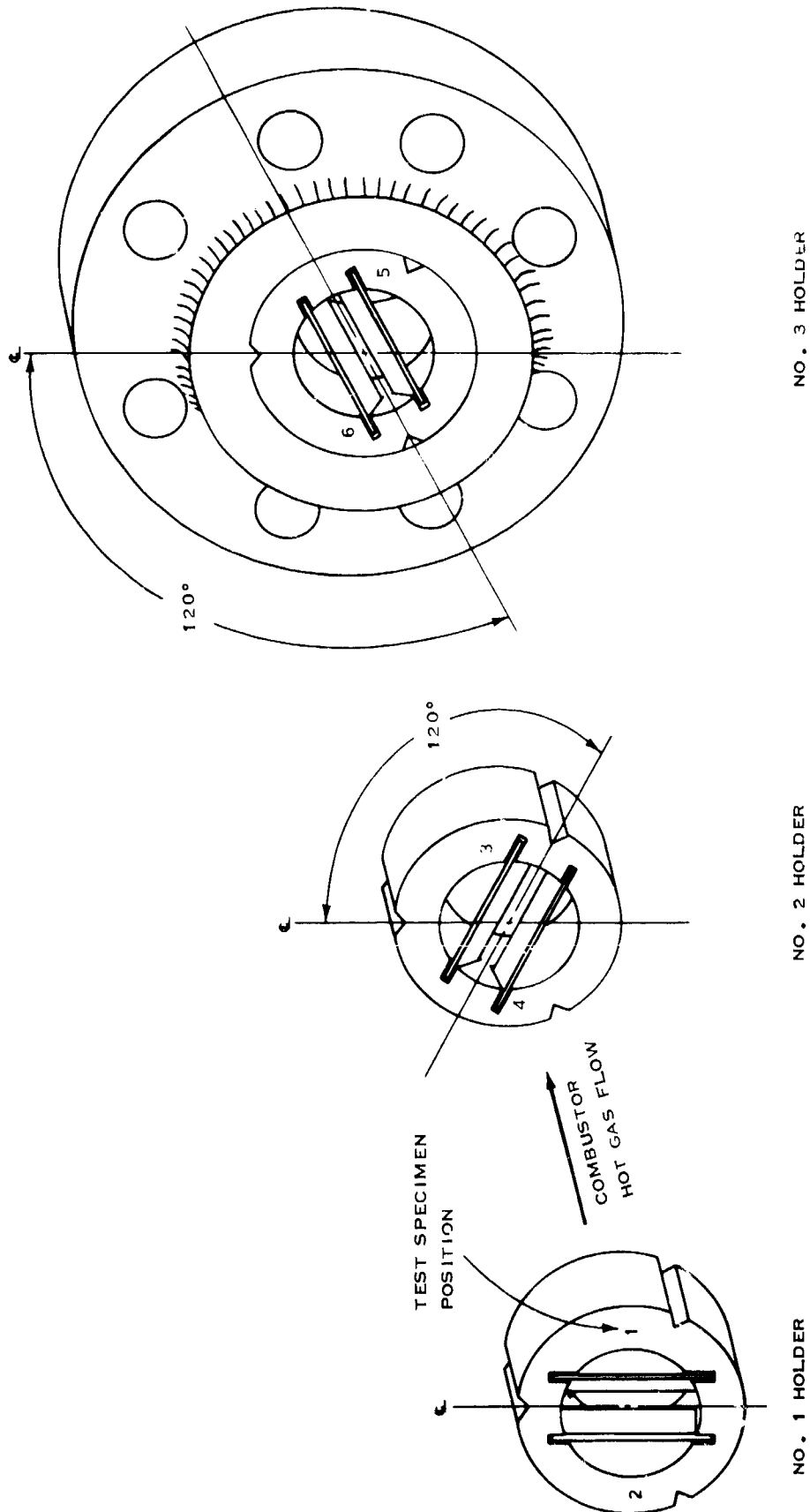


FIGURE 21  
CASCADE ASSEMBLY FOR TEST SPECIMENS



2X MAGNIFICATION

FIGURE 22  
SPECIMENS MOUNTED IN CASCADE

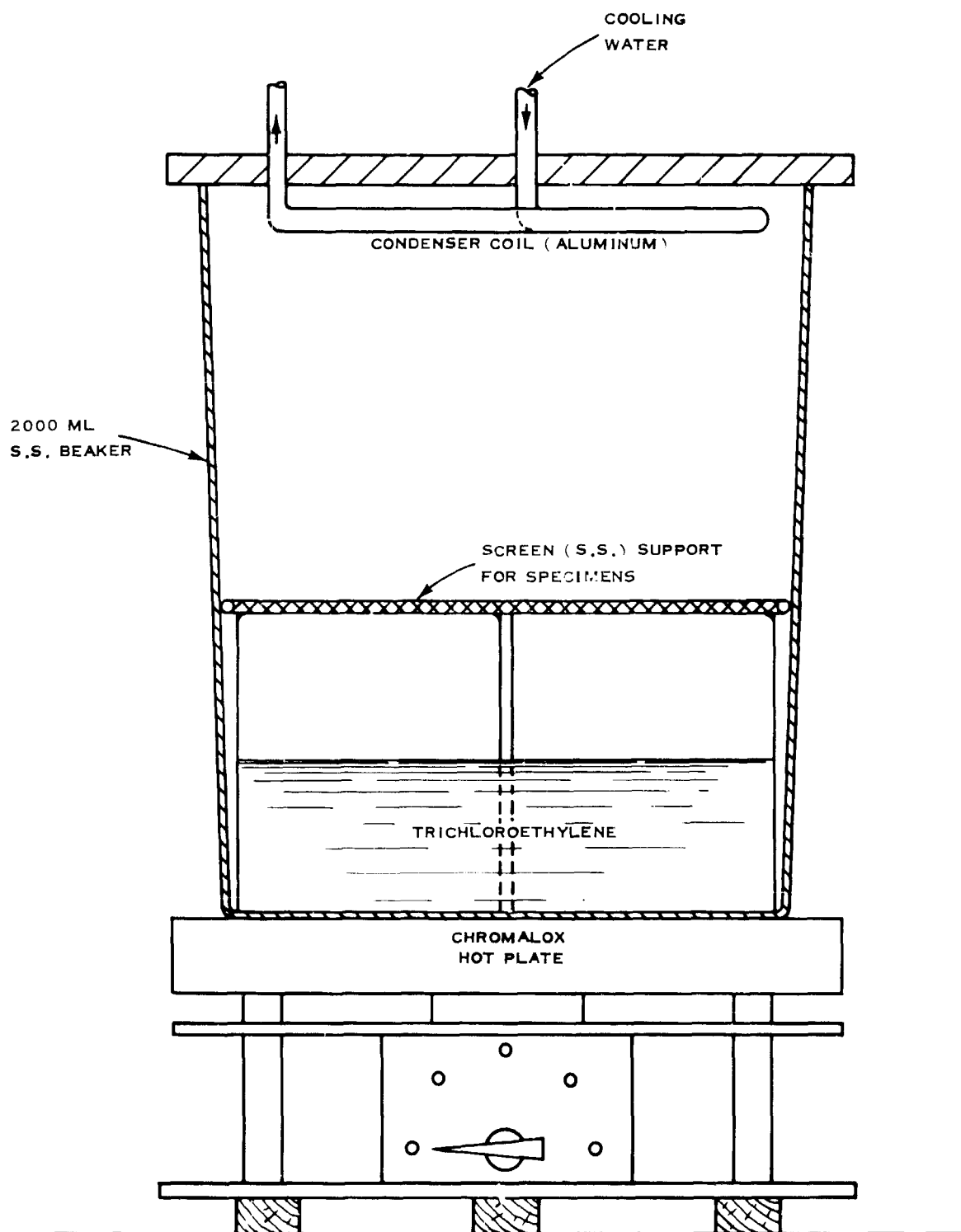


FIGURE 23  
VAPOR-DEGREASING APPARATUS FOR PRE-TEST CLEANING OF SPECIMENS

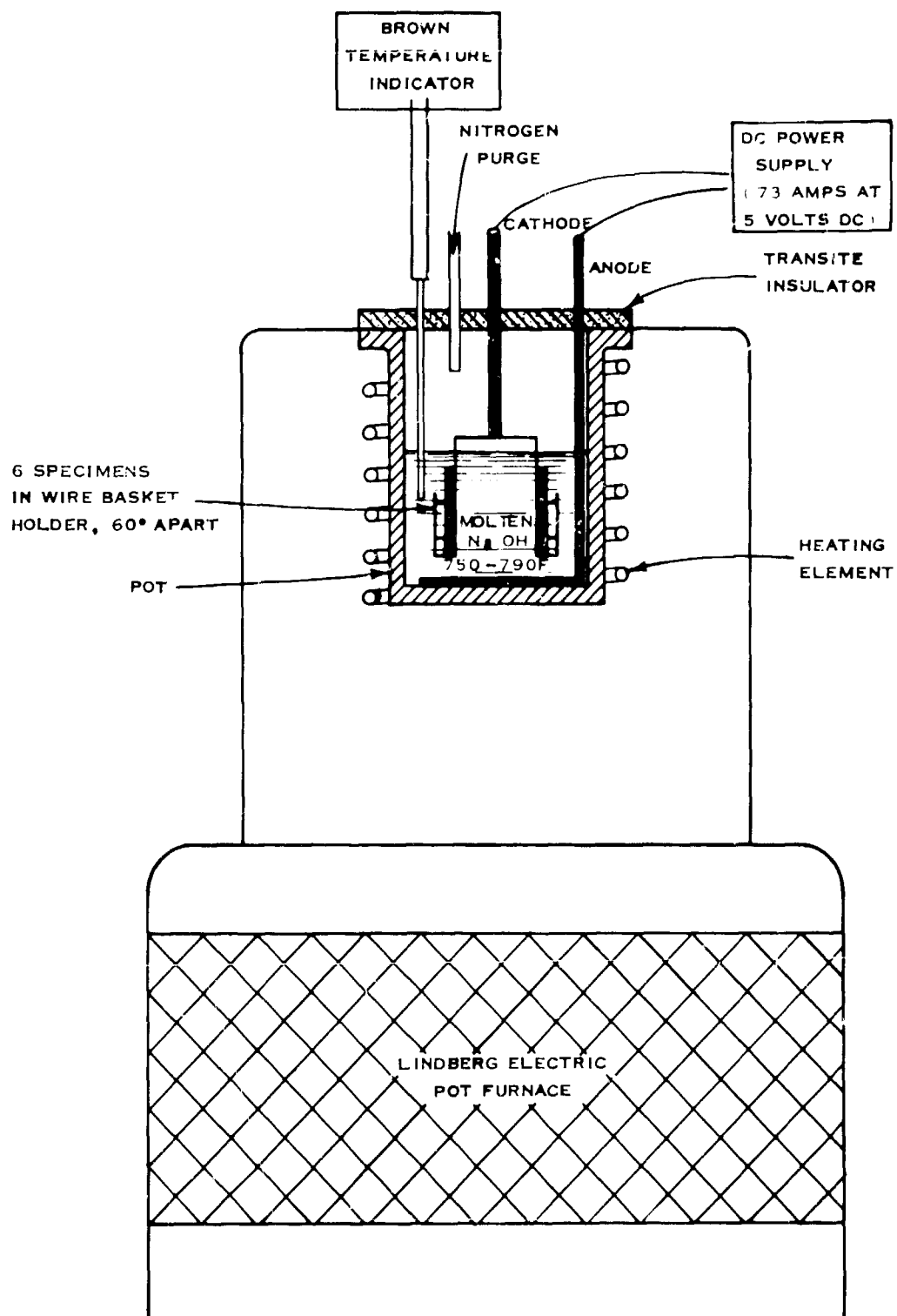


FIGURE 24  
ELECTRO-DESCALING APPARATUS  
FOR POST-TEST CLEANING OF SPECIMENS

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cleaning-loss with unexposed specimens approximates the weight-loss obtained by extrapolation of the curves shown in Figures 3-6 to zero hours of exposure. It compares with the cleaning-loss for unexposed Misco MDC-1 coated Inconel 713C specimens which averaged 1.2 mg/cm<sup>2</sup>, but which were cleaned only once (2).

#### 8.5. Specimen Examination

Following exposure, cleaning, and determination of weight-loss, the specimens were visually inspected. The system used for obtaining visual ratings is shown in Table 7; and the photographic standards used for rating surface and edge attack on the specimens are shown in Figures 25 and 26, respectively. The visual ratings are reported along with the weight-loss data.

Subsequently, specimens were chosen from each test for further metallographic examination. The selection was made to allow study of the effect of long-time exposure on the coating-alloy system at the 2000 F test condition, as well as changes in the concentration of corrosive agents which characterized the six different tests. This included Misco MDC-1 coated Mar M-200 specimens which had been exposed for 50 hours, if available, and in some cases at other exposure times where visual inspection indicated an unusual change in the mode or intensity of attack.

Each specimen was sectioned in three places, with one section at the point of maximum visible attack. The three pieces were placed in a 1-inch red Bakelite mold with the coupon of most interest (maximum attack) in the center. This arrangement aided in obtaining a flat surface during polishing. The following eight step technique was used to polish the coupons.

- (a) Dry ground on an 8", 60 grit Carbimet disc turned at 570 rpm.
- (b) Dry ground on an 8", 120 grit Carbimet disc turned at 570 rpm.
- (c) Dry ground on an 8", 180 grit Carbimet disc turned at 570 rpm.
- (d) Dry ground on an 8", 240 grit Carbimet disc turned at 570 rpm.
- (e) Hand lapped, wet, on 240 grit silicon carbide paper.
- (f) Hand lapped, wet, on 320 grit silicon carbide paper.
- (g) Hand lapped, wet, on 400 grit silicon carbide paper.
- (h) Hand lapped, wet, on 600 grit silicon carbide paper.
- (i) 6-micron diamond paste on nylon lap with polishing oil.
- (j) AB micro cloth with Linde B polishing compound.

Polishing was done in one direction. The edge of most interest was kept on the back side while polishing, so the polishing motion is across the coupons toward the edge to be kept flat.

TABLE 7

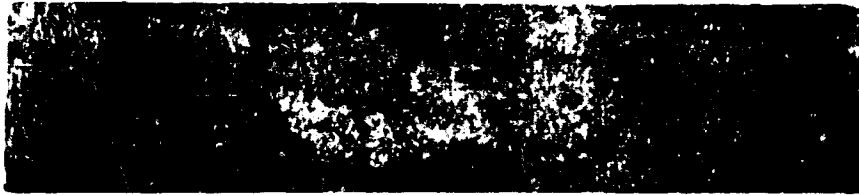
SYSTEM FOR VISUAL RATING OF ATTACK ON COATED TEST SPECIMENS

<u>Severity of Attack</u>	<u>Rating Code</u>	<u>Rating Value</u>
None	N	10
Very Light	VL	8
Light	L	6
Medium	M	4
Heavy	H	2
<u>Nature of Attack</u>		
Surface	S	
Edge	E	
<u>Condition of Specimen</u>		
Surface Cracks	C	

Example:

VLS = Very Light Surface Attack

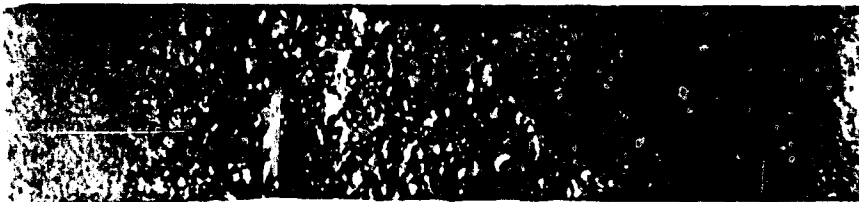




VERY LIGHT



LIGHT



MEDIUM



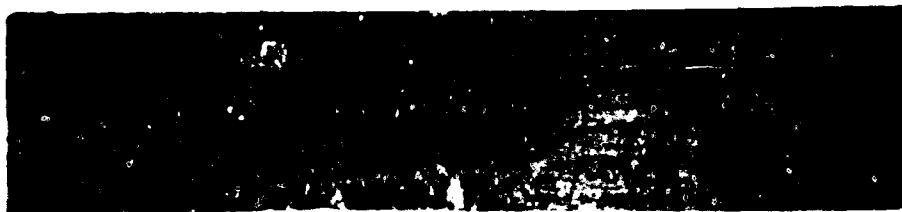
HEAVY

2X MAGNIFICATION

FIGURE 25  
STANDARDS FOR VISUAL RATING OF SURFACE ATTACK  
ON COATED SPECIMENS



VERY LIGHT



LIGHT



MEDIUM



HEAVY

2X MAGNIFICATION

FIGURE 26  
STANDARDS FOR VISUAL RATING OF EDGE ATTACK  
ON COATED SPECIMENS

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Appendix 1

In general, the attack was most evident on the impact surface of the specimens, as expected, because it had maximum exposure to the corrosive agents. Photomicrographs showing typical attack on the specimens were taken there at both 500 X and 2000 X, and in some cases at other locations and magnifications. Coupons were frequently etched to show areas of alloy depletion and grain structure. It is considered neither necessary nor desirable to reproduce in this report the eighty-one photomicrographs taken during our metallographic examination. Rather, typical photomicrographs are presented to illustrate the nature of the attack experienced by the coating and base alloy over the range of conditions investigated.

9. APPENDIX 2  
(Test Materials)

9.1. Test Fuels

9.1.1. Sulfur in JP-5

The sulfur content of 197 samples of grade JP-5 aviation turbine fuel, representative of domestic and foreign purchases by the United States Navy from September 1966 to February 1967 had a median value of 0.04 weight per cent. The cumulative distributions for West Coast, East and Gulf Coasts, foreign, and total are shown in Figure 27. These data indicate that a substantial, order-of-magnitude, reduction in the sulfur limit for JP-5 fuel could drastically curtail availability, unless accompanied by incentives for modernization of manufacturing techniques.

In previous investigations (1) three fuels were used having successive, order-of-magnitude, reductions in sulfur concentration, starting at the JP-5 sulfur limit; i.e., 0.40, 0.040, and <0.0040 weight per cent sulfur. These three levels of sulfur concentration span the range for samples in the survey, with the intermediate level of 0.040 weight per cent at the median value. It was decided that the current investigation would be conducted using the same three sulfur concentrations in fuel.

9.1.2. Base Fuel

The base fuel selected for use in this investigation was a segregated sample of production ASTM Type A aviation-turbine fuel. The physical and chemical properties of interest to this investigation are presented in Table 8. The average values of pertinent properties from the Bureau of Mines Product Survey (8) over the period of the past ten years are also shown for grade JP-5 aviation turbine fuel. The physical and chemical properties of the base fuel closely approximate the average for JP-5, with the exception of its very low sulfur content. The base fuel also was analyzed for metal content, to be certain that its iron, vanadium, nickel, and copper contents were negligible; if present, they would concentrate as ash and might alter the scale composition on the test specimens exposed to the exhaust gases.

The base fuel is essentially free of sulfur, containing less than 0.0040 weight per cent. Two fuels of higher sulfur contents were produced by blending to 0.040 and 0.40 per cent by weight of sulfur using ditertiary butyl disulfide.

9.2. Sea Water

9.2.1. Composition

A synthetic sea water was used in this study. Its formulation was taken from the Standard Method of Test for Rust-Preventing Characteristics

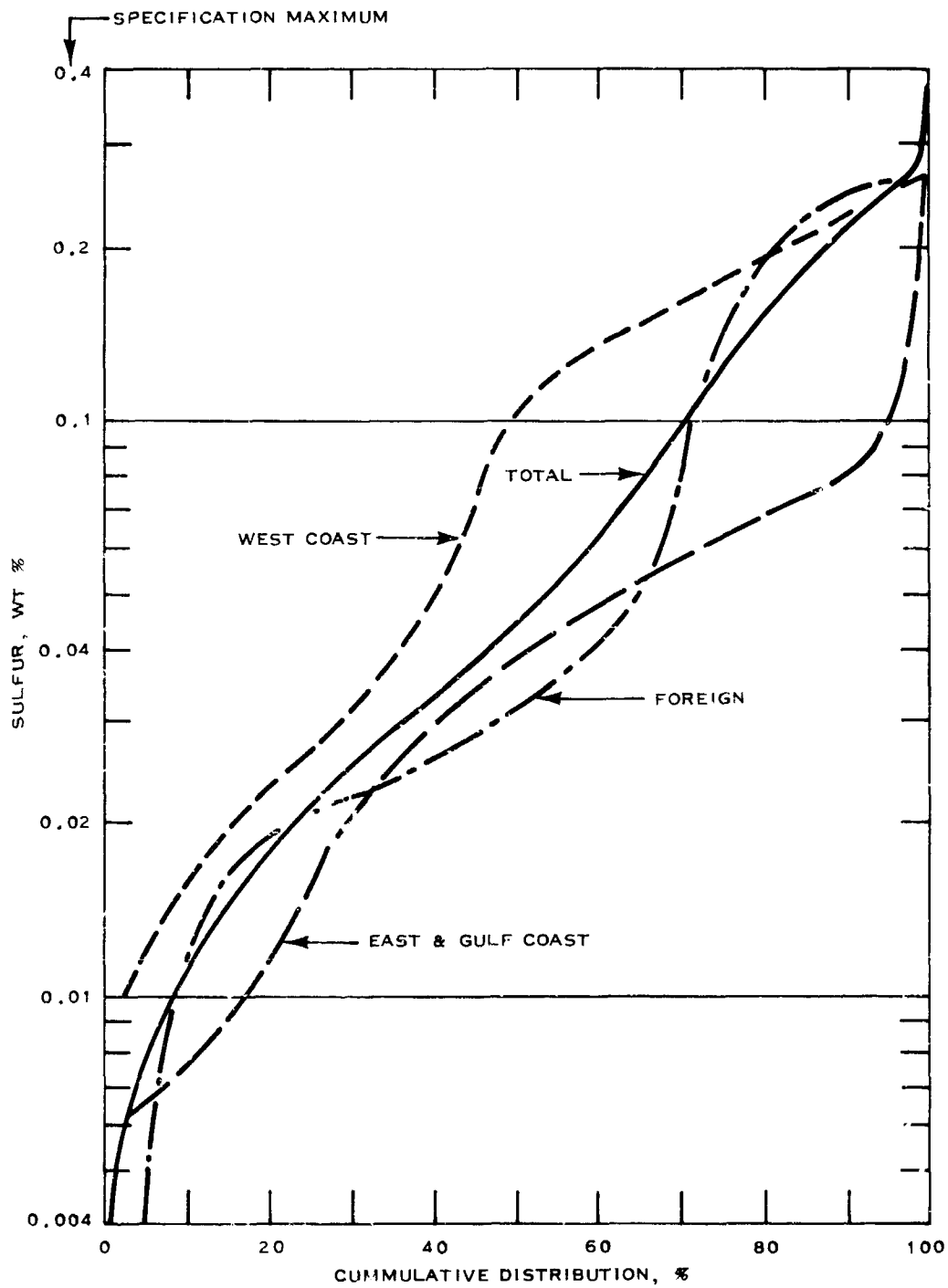


FIGURE 27  
SULFUR CONTENT OF JP-5 FUEL PURCHASES  
(SEPTEMBER 1966 - FEBRUARY 1967)

TABLE 8  
PHYSICAL AND CHEMICAL PROPERTIES OF TEST FUEL

	<u>Test Fuel</u> <u>Base (a)</u>	<u>Average</u> <u>JP-5 (b)</u>
Distillation Temperature, F		
Initial Boiling Point	326	...
5 volume per cent evaporated	352	...
10 volume per cent evaporated	357	...
20 volume per cent evaporated	358	382
30 volume per cent evaporated	377	...
40 volume per cent evaporated	388	...
50 volume per cent evaporated	398	413
60 volume per cent evaporated	410	...
70 volume per cent evaporated	423	...
80 volume per cent evaporated	438	...
90 volume per cent evaporated	457	455
95 volume per cent evaporated	473	...
End Point	493	...
Gravity, degrees API	45.9	41.9
Gum, milligrams per 100 mls.	0.5	1.1
Smoke Point, millimeters	26.2	22.6
Hydrogen Content, weight per cent	14.0	13.6
Composition, weight per cent		
Sulfur	< 0.0040 (c)	0.103
Metals		
Iron (d)	< 0.0001	...
Vanadium (d)	< 0.0002	...
Nickel (d)	< 0.0001	...
Copper (e)	0.0000037	...
Hydrocarbon Types volume per cent		
Normal Paraffins	27 (f)	...
Isoparaffins	23 (f)	...
Cycloparaffins	36 (f)	...
Olefins	0.33	1.6
Aromatics	13.37	15.1

Notes:

- (a) Segregated sample (BJ66-8-G7) of production ASTM Type A aviation turbine fuel, processed from West Texas crude and finished by hydrotreating.
- (b) U. S. Bureau of Mines Petroleum Product Survey, 1957 - 1966 (8).
- (c) Higher sulfur content test fuel obtained by blending to desired sulfur level using ditertiary butyl disulfide.
- (e) Spectro-photometric analysis.
- (f) Typical value for this product.

of Steam-Turbine Oil in the Presence of Water, ASTM Designation D-665-60. The components and their concentrations are shown in Table 9.

As discussed in Reference 1, the abundance of various elements in the synthetic formula compares very favorably with the average sea water composition. The one exception is silicon, and its exclusion from the synthetic sea water seems justified in the light of its reported variation in abundance from one water-mass to another by a factor of 1000, or more.

#### 9.2.2. Ingestion Rate

As discussed in Reference 1, establishing a realistic level for the concentration of sea salt in the air ingested by a gas-turbine engine operating in a marine environment is difficult from available literature. However, it was concluded that an ingestion rate of 1.0 ppm sea salt in air was a realistic level. Two concentrations of sea salt in air (zero and 1.0 ppm sea salt in air) were selected for use in the current investigation.

#### 9.3. Test Specimens

Previous studies (1,2) have been conducted to investigate the effect of sulfur in fuel on hot corrosion of bare and coated superalloys in a marine environment. In Reference 1, using 5-hour tests, five superalloys and one of the superalloys with a coating were included in the evaluation. It was found that a reduction in fuel sulfur by an order of magnitude (0.40 to 0.040 weight per cent) did not significantly reduce hot corrosion of a single alloy and with two superalloys under some conditions metal loss increased. The one coated superalloy (Misco MDC-1 on Inconel 713C) was immune to attack under all conditions of test. In Reference 2, using extended tests of up to 55-hours, with two coating alloy systems (Misco MDC-1 and MDC-9 on Inconel 713C) in the investigation it was found that an order of magnitude (0.40 to 0.040 weight per cent) reduction in fuel sulfur did not reduce hot corrosion significantly. It was concluded that a reduction in the specification maximum for fuel sulfur to reduce hot corrosion was not indicated on the basis of the bare and coated superalloys evaluated; however, it was recommended that additional coating-alloy systems be included in the evaluation.

In Reference 1, Inconel 713C (the base alloy for coatings in Reference 2) was not affected by a reduction in fuel sulfur while Mar M-200 was a superalloy that showed an increase in hot corrosion with a reduction in fuel sulfur. It was decided to select as a coating-alloy system for the current investigation a coating previously evaluated (Misco MDC-1) and use a base alloy that had shown a difference in performance in the bare condition. Therefore, the coating alloy system selected for use in the current investigation was Misco MDC-1 coated Mar M-200.

TABLE 9  
COMPOSITION OF ASTM D665 SYNTHETIC SEA WATER

<u>Salt (a)</u>	<u>Formula</u>	<u>Grams per liter (b)</u>
Sodium Chloride	NaCl	24.54
Magnesium Chloride	MgCl <sub>2</sub> ·6H <sub>2</sub> O	11.10
Sodium Sulfate	Na <sub>2</sub> SO <sub>4</sub>	4.09
Calcium Chloride	CaCl <sub>2</sub>	1.16
Potassium Chloride	KCl	0.69
Sodium Bicarbonate	NaHCO <sub>3</sub>	0.20
Potassium Bromide	KBr	0.10
Boric Acid	H <sub>3</sub> BO <sub>3</sub>	0.03
Strontium Chloride	SrCl <sub>2</sub> ·6H <sub>2</sub> O	0.04
Sodium Fluoride	NaF	<u>0.003</u>
	TOTAL	41.953

(a) Use cp chemicals.

(b) Use distilled water.



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Appendix 2

The Mar M-200 test specimens for this investigation were cast by the Misco Division, Howmet Corporation. The chemical analysis furnished by the supplier for the heat from which the specimens were cast is shown in Table 10.

The Misco MDC-1 coating was applied by the supplier to test specimens for Mar M-200. The coating-superalloy system is characterized by the following description.

MDC-1 is an aluminum coating, which was applied by a pack-diffusion process to obtain a total thickness of approximately 2 mils. It is divided about equally between an outer layer which contains non-metallic dispersions, and a diffused zone.

The investment castings of Mar M-200 were finished by the supplier by grinding to provide specimens having a smooth uniform surface with a thickness of  $0.125 \pm 0.005$  inches, a width of  $0.500 \pm 0.030$  inches and a length of  $2.375 \pm 0.030$  inches. The specimens were inspected by fluorescent penetrant (Zyglo) and X-ray to insure freedom from cracks, porosity and inclusions.

From physical measurements of a random sample of specimens the average surface area was calculated to be 20.27 square centimeters. From a random sample of Misco MDC-1 coated Mar M-200 specimens the average specimen weight was determined to be 20,915 mg.

Variability of weight-loss data with Misco MDC-1 coated Mar M-200 specimens in the current program was larger than experienced with coating-alloy systems tested previously. To obtain an estimate of repeatability of weight-loss data for comparison with data obtained previously, reference specimens were mounted at the rear of the cascade during one test with 0.4 weight per cent sulfur in fuel and 1.0 ppm sea salt in air. A new reference specimen was installed at each 5-hour period to give nine specimens for evaluation of variance.

Bare Inconel 713C specimens used in previous investigations (1, 2) had shown considerable loss of weight in a 5-hour period and, since specimens were available, Inconel 713C was selected as the reference material. Investment cast Inconel 713C specimens from Misco heat No. RW072 (2) were cut longitudinally to provide specimens with nominal dimensions of 1/8 inch thickness, 1/4 inch width and 2 3/8 inch length. The average surface area was calculated from physical measurements of the specimens to be 10.99 square centimeters.

TABLE 10

COMPOSITION OF MAR M-200 INVESTMENT CAST TEST SPECIMENS

<u>Alloying Elements</u>	<u>Chemical Analysis, Per Cent (a)</u>
Nickel	Balance (60.1)
Cobalt	0.1
Chromium	9.07
Molybdenum	...
Tungsten	12.2
Aluminum	4.92
Titanium	2.05
Manganese	0.1
Iron	0.19
Zirconium	0.042
Vanadium	...
Silicon	0.05
Boron	0.016
Sulfur	0.005
Carbon	0.15
Phosphorus	...
Copper	0.1
Cb + Ta	...
Cb	1.10

(a) Misco Heat Number RL088

10. APPENDIX 3  
(Test Program)

Previous tests (1) have shown that Misco MDC-1 coating on Inconel 713C was immune to attack in the standard 5-hour test over a range of temperatures and concentrations of sea salt in air and sulfur in fuel. However, it was shown (9) that test severity could be increased to the point of coating failure by extending the duration of the tests. Evaluation of two coating-alloy systems (Misco MDC-1 and MDC-9 on Inconel 713C) have been completed (2) which show one case where a reduction of sulfur in fuel from the present limit to 0.04 weight per cent significantly decreased hot corrosion, and another case where the attack was significantly increased. The objective of this experiment was to extend the evaluation of the effect of sulfur in fuel on hot corrosion in a marine environment to another coating-alloy system (Misco MDC-1 on Mar M-200).

The Phillips Test Facility and the Phillips 2-inch Combustor used for these experiments are described in Section 8.1. and 8.2. of Appendix 1. The 2000 F test condition was selected from previous studies (1), and is described in Table 11. Preparation of specimens for weighing, before and after exposure, is described in Section 8.4. of Appendix 1. The composition of the fuel used for this experiment is described in Section 9.1. of Appendix 2, composition of the synthetic sea water in Section 9.2., and composition of test specimens in Section 9.3.

In all tests the three stages of the cascade (Figure 21) were loaded with six new specimens prior to the start of a test. The cyclic test was operated in periods of 5-hours with the fuel nozzle, combustor dome and liner, sea water injection tubes and test specimens examined at the end of each period. The test plan for exposure variables is shown in Table 12. The basic plan for the experiment consisted of removal of one test specimen, with replacement, at each 5-hour period for the first five periods and then continuing the test to 55 hours, or until visual inspection of the specimens indicated heavy attack and the possible loss of a specimen with further exposure to the hot gases. To minimize any effect of position in the cascade, a system of rotation of the position of specimens was used where the specimen holder in the first stage of the cascade was moved to the third stage and the other two holders advanced one position at each five hour interval. The order of specimen removal is shown in Table 13. The scatter of weight-loss data in the presence of 1.0 ppm sea salt in air was greater than expected and additional tests were conducted with < 0.0040 and 0.40 weight per cent sulfur in fuel and 1.0 ppm sea salt in air. The plan for specimen removal was revised for the additional tests to provide for removal of specimens with 15, 30, or 45 hours of exposure and thus equal exposure time in each of the three stages of the cascade. With the revised schedule of specimen removal three specimens were available at each of the three periods of exposure and provided a better estimate of experimental error than with the previous schedule. The revised plan for specimen removal is shown in Table 14.

TABLE 11  
OPERATING CONDITIONS FOR PHILLIPS 2-INCH COMBUSTOR

<u>Test Variables</u>	<u>Test Conditions</u>
Temperature, deg. F	
Nominal Gas	2000
Exhaust Gas (a)	2039
Test Specimen, metal (b)	1970
Test Specimen, surface (c)	2074
Combustor Inlet Air	1000
Pressure, atmospheres	
Combustor Inlet Air	15
Mass Flow Rate, Pounds per hour	
Air	7200
Fuel	120
Air-Fuel Ratio	60
Flow Velocity, feet per second	
at Test Specimen (d)	745
Test Duration, hours (e)	Varied

Notes:

- (a) Values calculated using mean specific heats (10) for 100 per cent combustion efficiency. These values are confirmed by linear regression with measured temperatures in Appendix I of Reference 1.
- (b) Calculated mean value from thermocouple measurements of strip temperature vs calculated gas temperature in Appendix I of Reference 1.
- (c) Calculated mean value from optical pyrometer readings vs calculated gas temperature shown in Appendix I of Reference 1.
- (d) Calculated value based on unblocked area in specimen holder of 1.59 square inches.
- (e) Operating cycle was 55 minutes at test conditions followed by 5 minutes with fuel off.

TABLE 12  
TEST PLAN FOR EXPOSURE VARIABLES

<u>Sulfur in Fuel, wt %</u>	<u>Sea Salt in Air, ppm</u>	<u>Order of Test</u>
< 0.0040	0	5
< 0.0040	1.0	2
0.040	0	4
0.040	1.0	6
0.40	0	3
0.40	1.0	1

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TABLE 13  
ORDER OF TEST SPECIMEN REMOVAL

<u>Test Period</u>	<u>Test Time, Hours</u>	<u>Test Specimen Location</u>					
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
1	5	1 <sub>5</sub>	(2 <sub>5</sub> )	3 <sub>5</sub>	4 <sub>5</sub>	5 <sub>5</sub>	6 <sub>5</sub>
2	10	(3 <sub>10</sub> )	4 <sub>10</sub>	5 <sub>10</sub>	6 <sub>10</sub>	1 <sub>10</sub>	2 <sub>5</sub>
3	15	5 <sub>15</sub>	(6 <sub>15</sub> )	1 <sub>15</sub>	2 <sub>10</sub>	3 <sub>5</sub>	4 <sub>15</sub>
4	20	(1 <sub>20</sub> )	2 <sub>15</sub>	3 <sub>10</sub>	4 <sub>20</sub>	5 <sub>20</sub>	6 <sub>5</sub>
5	25	3 <sub>15</sub>	(4 <sub>25</sub> )	5 <sub>25</sub>	6 <sub>10</sub>	1 <sub>5</sub>	2 <sub>20</sub>
6	30	5 <sub>30</sub>	6 <sub>15</sub>	1 <sub>10</sub>	2 <sub>25</sub>	3 <sub>20</sub>	4 <sub>5</sub>
7	35	1 <sub>15</sub>	2 <sub>30</sub>	3 <sub>25</sub>	4 <sub>10</sub>	5 <sub>35</sub>	6 <sub>20</sub>
8	40	3 <sub>30</sub>	4 <sub>15</sub>	5 <sub>40</sub>	6 <sub>25</sub>	1 <sub>20</sub>	2 <sub>35</sub>
9	45	5 <sub>45</sub>	6 <sub>30</sub>	1 <sub>25</sub>	2 <sub>40</sub>	3 <sub>35</sub>	4 <sub>20</sub>
10	50	1 <sub>30</sub>	2 <sub>45</sub>	3 <sub>40</sub>	4 <sub>25</sub>	5 <sub>50</sub>	6 <sub>35</sub>
11	55	3 <sub>45</sub>	4 <sub>30</sub>	5 <sub>55</sub>	6 <sub>40</sub>	1 <sub>35</sub>	2 <sub>50</sub>

Notes:

- ( ) Remove test specimen and replace with new test specimen.  
Terminate test when failure of a specimen appears imminent.

TABLE 14  
REVISED PLAN FOR ORDER OF TEST SPECIMEN REMOVAL

<u>Test Period</u>	<u>Test Time, hours</u>	<u>CASCADE POSITION</u>					
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
		<u>Test Specimen Location</u>					
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
1	5	1 <sub>5</sub>	2 <sub>5</sub>	3 <sub>5</sub>	4 <sub>5</sub>	5 <sub>5</sub>	6 <sub>5</sub>
2	10	3 <sub>10</sub>	4 <sub>10</sub>	5 <sub>10</sub>	6 <sub>10</sub>	1 <sub>10</sub>	2 <sub>10</sub>
3	15	5 <sub>15</sub>	(6 <sub>15</sub> )	1 <sub>15</sub>	(2 <sub>15</sub> )	3 <sub>15</sub>	4 <sub>15</sub>
4	20	1 <sub>20</sub>	2 <sub>50</sub>	3 <sub>20</sub>	4 <sub>20</sub>	5 <sub>20</sub>	6 <sub>5</sub>
5	25	3 <sub>25</sub>	4 <sub>25</sub>	5 <sub>25</sub>	6 <sub>10</sub>	1 <sub>25</sub>	2 <sub>10</sub>
6	30	5 <sub>30</sub>	6 <sub>15</sub>	1 <sub>30</sub>	2 <sub>15</sub>	(3 <sub>30</sub> )	4 <sub>30</sub>
7	35	1 <sub>35</sub>	2 <sub>20</sub>	3 <sub>5</sub>	4 <sub>35</sub>	5 <sub>35</sub>	6 <sub>20</sub>
8	40	3 <sub>10</sub>	4 <sub>40</sub>	5 <sub>40</sub>	6 <sub>25</sub>	1 <sub>40</sub>	2 <sub>25</sub>
9	45	5 <sub>45</sub>	6 <sub>30</sub>	1 <sub>45</sub>	2 <sub>30</sub>	3 <sub>15</sub>	4 <sub>45</sub>

( ) Remove specimen and replace with new specimen.

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For one test, a reference specimen of bare Inconel 713C was mounted at the rear of the cascade. This specimen was replaced with a new specimen at the end of each 5-hour period to provide nine specimens for evaluation of test rig repeatability.



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11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Department of the Navy Naval Air Systems Command Washington, D. C. 20360
13. ABSTRACT Specimens of a nickel-base alloy (Mar M-200) with an aluminum-diffusion coating (Misco MDC-1) were tested using a high-pressure combustor facility to simulate environment in the turbine section of an aircraft engine. Tests were conducted at 2000 F gas temperature, without sea salt and with 1.0 ppm sea salt in air, using three levels of sulfur in fuel (0.40, 0.040 and <0.0040 per cent by weight. A significant decrease in the relative rate of corrosion and the level of attack when sea salt was removed from the air indicated that sea salt is a primary cause in the hot corrosion of both bare and coated superalloys in a marine environment. The effect of sulfur in fuel varied with the absence or presence of sea salt. In the absence of sea salt, a reduction in sulfur from the present limit of 0.40 per cent for JP-5 fuel to 0.040 per cent, or less, decreased hot corrosion; however, the level of attack was so low that the reduction may be of little practical significance. In the presence of 1.0 ppm sea salt in air, a reduction in sulfur to either 0.040 or <0.0040 per cent had no statistically significant effect on hot corrosion; but the experimental error was considerably greater with this coating-alloy system than with other systems evaluated that benefited from a reduction in sulfur to less than 0.0040 per cent.		

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